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Notes

The Ascorbic Acid and Carotene Content of some Australian Fruits and Vegetables*

1. Introduction

From 1942 to 1945 a considerable number of ascorbic acid determinations were carried out in the laboratories of Commonwealth Food Control and the Council for Scientific and Industrial Research. These determinations were made in connexion with the supply of food to the Services, and were directed primarily to ensuring maximum retention during processing. Some determinations of carotene were made during the same period. These data may be of general interest in the field of nutrition, and are here presented.

Determinations of ascorbic acid by Commonwealth Food Control were made in every State. In addition, the Division of Food Preservation and Transport of the Commonwealth Council for Scientific and Industrial Research carried out many ascorbic acid determinations in the laboratory at Homebush, New South Wales. All the carotene determinations were made in this laboratory. Additional ascorbic acid data were obtained in Tasmania by the Division of Plant Industry, Council for Scientific and Industrial Research.

2. Procedure

Ascorbic acid was determined by the usual procedure, i.e. extraction with 3 per cent. metaphosphoric acid and titration with 2, 6-dichlorophenolindophenol. Carotene was determined by the procedure of Austin and Shipton (1944)†.

The data are presented in Tables 1 to 5. Both ascorbic acid and carotene are expressed as mg. per 100 g. (The usual procedure is to regard 1 mg. of ascorbic acid as equivalent to 20 International Units (I.U.) of vitamin C, and 1 mg. of β-carotene as equivalent to 1667 I.U. of vitamin A.) In many cases, the tables provide all the essential information, but for some products additional notes are provided in the text.

The data given in the tables represent, as far as possible, random samples of each product. In most cases the figures are for commercial material which has been received for examination or fresh material for processing experiments. It should be approximately

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representative of material passing into consumption during the period
of the determinations. All the processed material was analysed soon
after processing. Subsequent storage often involves some loss of
vitamin. Data derived from variety trials are generally excluded from
this paper. Such trials usually involved only a few determinations
for each of a large number of varieties, many of which are only of
minor importance commercially and cannot be regarded as
representative.

The tabulated data refer to the portion of the product which is
normally consumed, such as the solid portion of canned foods (except
juices). The individual determinations are quoted where not more
than ten samples were analysed. Otherwise, the data are summarized
by giving the mean and standard deviation (S.D.).

In almost all cases where the mean and standard deviation are
quoted the distribution of the values was approximately normal.
Among the important consequences of this are that the mean can be
taken as the most representative value and that approximately 95 per
cent. of the values fall in the range—Mean $\pm 2 \times$ S.D.

Cases where the distribution departed markedly from the normal
form are indicated in the text. Most of the dehydrated products
gave non-normal, flat-topped distribution curves. In such cases the
total range would be over-estimated if normality is assumed.

Significant differences between States appear in some products.
The origin of these differences is usually uncertain, as it involves
many factors such as climate, soil, cultural practices, and variety.
With processed foods, processing practice may have differed sig-
nificantly from State to State.

The maximum percentage moisture in the dried products is as
follows: cabbage, 4; carrots, 5; potatoes, 7; silver beet, 4.

3. Ascorbic Acid in Fruit Products (Tables 1, 2, and 3)

(1) *Apples.*

In a variety trial of fresh apples in Tasmania in 1944, the Sturmer
variety was found to have a much higher ascorbic acid content than
any other of the main varieties. Following are the figures for ascorbic
acid (as mg./100g.) in whole fruit (including skin).

Alfriston—11.
Cleopatra—14.
Cox's Orange Pippin—9, 12, 12.
Crofton—11.
Delicious—9, 11.
Democrat—11, 13.
French Crab—6, 14, 15.
Granny Smith—10.
Jonathan—11.
Sturmer (17 samples)—Mean 25·5, S.D. 1·1.

The data for canned apples (mean 8·5, S.D. 4·2) are derived from
a skew distribution. In this case the median is 7·9 and the range
0·5–18·6.

(ii) *Blackcurrant Products.*

Neither canned fruit nor canned pulp are normally consumed as such. Unsweetened blackcurrant is excessively sour, and hence this fruit is usually consumed as jam or syrup. The bulk of the blackcurrant pulp is processed again into jam or syrup.

(iii) *Grapefruit Juice, Canned.*

The figures do not differ significantly from State to State.

(iv) *Lemon Juice, Canned.*

The figures for New South Wales were above, and those for Queensland were below, the average for all the States.

(v) *Orange Products.*

The figures for canned orange juice show a very definite monthly variation (Table 2). There is a gradual decline from August to February in all States except Queensland. In the latter State, common oranges predominated in the varieties processed and the season practically finished at the end of September.

This decline is almost certainly due to the seasonal decline in late Valencia oranges. Except in Queensland, Navel oranges predominated before and Valencia oranges after September. This has been shown by studying the monthly variation in ascorbic acid content of fresh oranges from the Commonwealth Research Station, Griffith, New South Wales, during 1944 and 1945.

The determinations were made about the middle of each month. The figures given in Table 3 represent the average for two years. The juice from Washington Navel oranges did not vary appreciably in ascorbic acid content during the usual picking period (May to September), but the late Valencia orange juice declined steadily in ascorbic acid from August to February. The Navel juice is generally most palatable in August and the Valencia juice in October.

Figures for inner and outer rind are given in Table 3. The inner rind includes " rag," i.e. pith and segment walls which are left after pressing out the juice. The ascorbic acid content of the rind increased during the season in Navels, but decreased considerably in Valencias. The outer rind was particularly high in ascorbic acid. In both varieties, maximum ascorbic acid was approximately associated with maximum orange colour of the rind. Although not eaten fresh, the rind can contribute quite an appreciable level of ascorbic acid to orange jam and marmalade. By taking precautions to reduce loss, Huelin and Stephens (1944)[*] have prepared " marmalade mixture " containing 20 mg. of ascorbic acid per 100 g. almost entirely from rind and " rag."

(vi) *Pineapple Juice, Canned.*

The figures for ascorbic acid are derived mostly from the Smooth Cayenne variety. The Ripley Queen, which is mostly consumed fresh, is more than twice as rich in ascorbic acid. The mean figures

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[*] Huelin, F. E., and Stephens, I. M. (1944).—*Aust. Food Manuf.* 13 (7): 2.

over the whole season do not give a complete picture, as there is quite an appreciable difference between the summer and winter crop.

Summer crop (Feb.-May): Mean 8·7, S.D. 1·6.

Winter crop (June-Oct.): Mean 13·1, S.D. 2·4.

4. Ascorbic Acid in Vegetable Products (Table 4)

(i) *Cabbage.*

The data for fresh cabbage given in Table 4 (mean 58·1, S.D. 13·0) are derived from a bimodal distribution with maximum about 58 and subsidiary peak about 35. These figures are for the portion usually consumed, i.e. with the outer green leaves removed. The outer portion is, however, definitely richer in ascorbic acid, as shown by the following determinations: 71, 91, 106, 108, and 133 mg./100 g.

Dried cabbage (mean 327, S.D. 110) gives a flat-topped distribution from 150 to 500 with median about 350.

(ii) *Peas, Green, Canned.*

The data from New South Wales give a flat-topped distribution with median of 11.

(iii) *Potatoes, Dried.*

The data for dried potatoes give non-normal, flat-topped distribution curves.

(iv) *Tomato Products, Canned.*

The tomato products show a slight seasonal variation, the mid-season (Feb.-March) samples being slightly higher in ascorbic acid than later (April-June) samples. The average difference is about 1 mg. per 100 g.

The figures from Victoria and Western Australia are significantly higher than those from New South Wales and South Australia. The Tasmanian figures are significantly lower than those from the other States.

5. Ascorbic Acid in Rose Hips

The analyses of rose hips are not given in the tables, which are confined to what are commonly regarded as fruit and vegetable products. Rose hips, however, are extremely high in ascorbic acid, and extracts can be added to other products to increase the ascorbic acid contents. The following data are available from three States:—

Victoria—453, 483, 495 mg./100 g.

New South Wales—517, 543, 553, 560 mg./100 g.

Tasmania—561, 652, 674, 780, 781, 816, 833 mg./100 g.

6. Carotene in Fruit and Vegetable Products (Table 5)

Carrots, Dried.—These figures should be used with caution, as they are derived by different processing techniques from a relatively small number of fresh samples.

TABLE 1.—ASCORBIC ACID IN FRUIT PRODUCTS.

Product.	State.	Number of Samples.	Ascorbic Acid (mg./100 g.).
Apples, Canned ..	Tasmania ..	31	Mean 8·5, S.D. 4·2
Blackcurrants, Canned ..	Tasmania ..	27	Mean 107·5, S.D. 13·6
Blackcurrant Pulp, Canned	Tasmania ..	217	Moan 153·4, S.D. 21·7
Gooseberries, Canned ..	Tasmania ..	33	Mean 24·0, S.D. 3·5
Grapefruit Juice, Canned*	Victoria ..	183	Mean 40·6
Grapefruit Juice, Canned*	New South Wales	131	Mean 39·6
Grapefruit Juice, Canned*	Queensland ..	21	Mean 40·4
Grapefruit Juice, Canned*	South Australia	29	Mean 39·1
Grapefruit Juice, Canned*	Total	364	Mean 40·1, S.D. 5·8
Lemon Juice, Canned* ..	Victoria ..	9	Mean 39·0
Lemon Juice, Canned* ..	New South Wales	201	Mean 42·7
Lemon Juice, Canned* ..	Queensland ..	63	Mean 34·6
Lemon Juice, Canned* ..	South Australia	31	Mean 38·8
Lemon Juice, Canned* ..	Total	304	Mean 39·2, S.D. 5·0
Loganberries, Canned ..	Tasmania ..	10	6, 9, 10, 12, 12, 13, 14, 15, 17, 21
Orange Juice, Canned* ..	Victoria ..	272	M .n 53·2, S.D. 9·7
Orange Juice, Canned* ..	New South Wales	1,000	Mean 55·4, S.D. 7·2
Orange Juice, Canned* ..	Queensland ..	205	Mean 56·0, S.D. 6·1
Orange Juice, Canned* ..	South Australia..	245	Mean 53·6, S.D. 5·0
Orange Juice, Canned* ..	Total	1,722	Mean 54·9, S.D. 7·3
Pineapple Juice, Canned*	Queensland ..	183	Mean 11·0, S.D. 3·0
Raspberries, Fresh ..	Tasmania ..	13	Mean 19·1, S.D. 6·4

* Ascorbic acid expressed as mg./100 ml.

TABLE 2.—MONTHLY VARIATION IN ASCORBIC ACID IN CANNED ORANGE JUICE.

State.	Month Canned.	Number of Samples.	Ascorbic Acid (mg./100 ml.).	
			Mean.	S.D.
Victoria	June	25	57·0	5·3
	July	21	58·2	3·6
	August	27	61·8	7·7
	September ..	21	61·5	5·8
	October	28	59·0	7·0
	November ..	36	55·8	5·5
	December· ..	34	52·7	5·6
	January	40	46·3	5·2
	February ..	38	38·4	5·2
	March	2	33·4	..
New South Wales ..	June	45	54·1	5·0
	July	117	54·9	6·2
	August	128	62·5	7·4
	September ..	94	56·5	8·3
	October	134	55·4	6·8
	November ..	191	56·2	5·9
	December ..	121	55·0	5·0
	January ..	142	50·4	5·0
	February ..	28	45·9	5·0
Queensland	May	21	56·8	5·1
	June	26	50·5	6·7
	July	61	54·5	6·1
	August	46	59·0	3·7
	September ..	41	57·2	3·4
	October	9	64·7	2·3
	November ..	1	68·4	..
South Australia .	June . ..	29	57·9	3·4
	July	44	55·3	2·2
	August	61	55·8	2·9
	September ..	46	53·7	4·2
	October	16	54·4	4·3
	November ..	21	50·1	3·7
	December ..	16	48·3	5·0
	January	9	45·1	6 4
	February ..	3	• 40·1	..

TABLE 3.—MONTHLY VARIATION IN ASCORBIC ACID IN FRESH ORANGES FROM GRIFFITH, NEW SOUTH WALES.

Variety.	Month Picked.	Ascorbic Acid in—		
		Juice (mg /100 ml.)	Inner Rind (mg /100 g.).	Outer Rind. (mg./100 g.).
Washington Navel ✓..	April	72	64	193
	May	67	65	211
	June	69	85	278
	July	68	84	268
	August	68	89	280
	September	74	125	325
Late Valencia ✓ ..	August	70	83	260
	September	67	84	236
	October	64	59	218
	November	61	56	216
	December	55	48	183
	January	51	40	141
	February	48	36	119

TABLE 4.—ASCORBIC ACID IN VEGETABLE PRODUCTS.

Product.	State.	Number of Samples	Ascorbic Acid (mg./100 g.).
Asparagus, Canned ..	New South Wales	10	10, 11, 12, 13, 14, 19, 19, 23, 23, 24
Cabbage, Fresh ..	New South Wales	77	Mean 58·1, S.D 13·0
Cabbage, Canned ..	New South Wales	65	Mean 13·9, S.D. 5·9
Cabbage, Canned ..	Tasmania	99	Mean 13·2, S.D. 2·5
Cabbage, Dried ..	New South Wales	94	Mean 327, S.D. 110
Carrots, Canned ..	New South Wales	18	Mean 3·3, S.D. 1·4
Parsnips, Canned ..	New South Wales	18	Mean 3·8, S.D. 2·9
Peas, Green, Canned ..	New South Wales	38	Mean 11·4, S.D. 7·7
Peas, Green, Canned ..	Tasmania	38	Mean 13·4, S.D. 1·6
Potatoes, Fresh ..	Victoria	34	
Potatoes, Fresh ..	New South Wales	20	
Potatoes, Fresh ..	Tasmania	48	
Potatoes, Fresh ..	Total ..	102	Mean 14·4, S.D. 6·0
Potatoes, Dried ..	Victoria	32	
Potatoes, Dried ..	New South Wales	21	
Potatoes, Dried ..	Tasmania	46	
Potatoes, Dried ..	Total ..	99	Mean 39·6, S.D. 23·0

TABLE 4.—ASCORBIC ACID IN VEGETABLE PRODUCTS—*continued*.

Product.	State.	Number of Samples.	Ascorbic Acid (mg./100g.)
Silver Beet, Fresh ..	New South Wales	10	26, 26, 28, 28, 29, 31, 32, 53, 36, 50
Silver Beet, Canned ..	New South Wa.es	16	Mean 8·5, S.D. 5·2
Silver Beet, Dried ..	New South Wales	10	78, 82, 101, 117, 121, 125, 132, 229, 261, 343
Tomatoes, Fresh ..	Tasmania ..	29	Mean 22·3, S.D. 4·9
Tomatoes, Canned ..	Victoria .	1,345	Mean 21·7, S.D. 3·1
Tomatoes, Canned ..	New South Wales	538	Mean 17·6, S.D. 3·7
Tomatoes, Canned .	South Australia..	106	Mean 18·0, S.D. 2·5
Tomatoes, Canned ..	Western Australia	112	Mean 22·9, S.D. 2·9
Tomatoes, Canned ..	Tasmania ..	198	Mean 13·9, S.D. 3·1
Tomatoes, Canned ..	Total	2,299	Mean 20·1, S.D. 4·1
Tomato Juice, Canned* ..	Victoria ..	1,545	Mean 20·1, S.D. 3·7
Tomato Juice, Canned* ..	New South Wales	834	Mean 17·3, S.D. 4·0
Tomato Juice, Canned* ..	South Australia..	63	Mean 18·0, S.D. 1·9
Tomato Juice, Canned* ..	Western Australia	228	Mean 20·2, S.D. 2·4
Tomato Juice, Canned* ..	Tasmania ..	21	Mean 13·7
Tomato Juice, Canned* ..	Total	2,691	Mean 19·2, S.D. 3·9
Tomato Pulp, Canned ..	Victoria ..	1,825	Mean 21·0, S.D. 4·3
Tomato Pulp, Canned ..	New South Wales	349	Mean 18·3, S.D. 4·5
Tomato Pulp, Canned .	South Australia..	143	Mean 14·9, S.D. 4·2
Tomato Pulp, Canned ..	Tasmania ..	209	Mean 13·8, S.D. 5·4
Tomato Pulp, Canned ..	Total ..	2,526	Mean 19·7, S.D. 5·1

* Ascorbic acid expressed as mg /100 ml.

TABLE 5.—CAROTENE IN FRUIT AND VEGETABLE PRODUCTS.

Product.	State	Number of Samples	Carotene (mg /100 g.)
Apricots, Dried ..	New South Wales	8	7·5, 7·7, 8·2, 8·8, 10·7, 10·8, 10·9, 11·0
Carrots, Fresh ..	New South Wales	13	Mean 15·07, S.D. 2·48
Carrots, Dried ..	New South Wales	81	Mean 135·0, S.D. 19·3
Silver Beet, Fresh ..	New South Wales	10	2·8, 4·6, 4·8, 5·1, 5·7, 5·7, 5·7, 5·8, 5·8, 6·2
Silver Beet, Dried ..	New South Wales	9	37·3, 41·8, 41·8, 43·7, 46·8, 51·9, 53·., 53·5, 54·8

A Survey of Insect Pests and Details of Insecticide Trials on Army Farms in the Northern Territory

By G. A. H. Helson, M.Sc.*

Summary.

A survey of insect pests attacking fruit and vegetable crops was made on Army farms in the Northern Territory during the " dry " season, 1945. Twenty-two insect pests and one nematode were identified attacking crops at this time. The most important pests were corn earworm, *Heliothis armigera* Hubn., centre grub, *Hellula undalis* Fabr., and cabbage moth, *Plutella maculipennis* Curtis.

Insecticidal experiments for the control of these pests were made using crude hexachlorocyclohexane (666), DDT, and synthetic cryolite. Some of these experiments could not be completed, but sufficient evidence was obtained to show that all of them were effective and that the most useful of these was DDT used as a spray or as a dust.

1. Introduction

In 1940 Army Farm Units of the Australian Army Service Corps acquired a neglected peanut farm consisting of 107 acres at Adelaide River, Northern Territory, and thus began a series of farms which eventually extended from Spinnifex, in Central Australia, to Coomallie Creek, in the Northern Territory. These farms were developed to meet the increasing demand by service personnel for fresh vegetables, fruit, and poultry which followed the progressive development of hostilities in the tropical regions of Australia and the islands to the north. A description of these farms and the crops they produced has been given by Kjar (1945).

The main farms were established at Adelaide River (130 acres), and at Katherine River (120 acres). The Katherine experiment farm was begun early in 1944, when 16 acres of virgin bush typical of the river fringe land was cleared and developed into the 1st Australian Experimental Farm by the 1st Australian Experimental Farm Platoon. This farm was established to aid agriculture in the Northern Territory, a policy which is being continued by the Council for Scientific and Industrial Research, which has acquired the farm.

The initial crops grown on the farms were not troubled by insect pests to any great extent, but as time went on, with new areas being brought under cultivation and old ones extended, the crops were attacked by endemic insects and particularly by pests introduced on fruit and vegetables imported for Service needs. In order to advise the personnel on the latest methods and insecticides for the control of insect pests, a survey was arranged, and this gave a unique opportunity of observing the pests, their effect on crops, and the methods of control in use. Many of the crops were being grown on a large scale in the Northern Territory for the first time. The survey was made during the middle of the " dry " season of 1945, at the height of the growing period, and while it is by no means complete, the survey does include all the principal pests occurring on crops which were grown at that time.

* An officer of Division of Economic Entomology.

As it is anticipated that many of the crops grown during the war will continue to be grown in the Northern Territory in the future, it seemed desirable that the results of the survey should be published for future reference. The present paper records these, and describes some field experiments carried out to test the effectiveness of DDT, crude hexachlorocyclohexane (666), and synthetic cryolite against the pests of cabbages and tomatoes, and to test the usefulness of a mixture of dichloropropane and dichloropropylene as a fumigant for the control of root-knot eelworm, *Heterodera marioni* (Cornu), which had become troublesome in seedbeds at Adelaide River.

2. Results of the Survey

The survey was made on all farms being worked in 1945 at Katherine River, Adelaide River, and Coomallie Creek. The insects which were collected and identified, and the hosts from which they were taken are set out in Tables 1, 2, and 3.

TABLE 1.—INSECTS RECORDED ATTACKING CROPS AT KATHERINE RIVER, NORTHERN TERRITORY—JULY, 1945.

Crop Attacked.	Insect Pest.	
	Scientific Name.	Common Name.
Beetroot ..	*Hellula undalis* Fabr. .. .. *Hymenia recurvalis* F.	Centre grub Beet webworm
Cabbage ..	*Heliothis armigera* Hubn. .. .. *H. undalis* .. .. .. *Plutella maculipennis* Curtis .. *Agrotis* sp.	Corn earworm Centre grub Cabbage moth Cutworm
French bean ..	*Agromyza phaseoli* Coq.	Bean fly
Grain sorghum ..	*H. armigera* .. .. .. *Aphis maidis* Fitch	Corn earworm Corn aphis
Lablab bean ..	Unidentified immature forms only ..	Bean leaf miner
Lettuce .	*Empoasca* sp. .. .. .. *Orosius argentata* (Evans) ..	Green jassid Brown jassid
Papaw	Coccidae, unidentified .. .. Sphingidae, unidentified larval form only	Scale insect Hawk moth
Potato .'. ..	*Gnorimoschema operculella* (Zell.) ..	Potato moth

TABLE 1—*continued.*

Crop Attacked.	Insect Pest	
	Scientific Name.	Common Name.
Pumpkin ..	*Epilachna 28-punctata*	Leaf-eating ladybird
	Ceratia hilaris Boisd.	Pumpkin beetle
	Rhaphidopalpa palmerstoni Blkb. ..	Pumpkin beetle
Sweet corn ..	*H. armigera*	Corn earworm
	A. maidis ..	Corn aphis
Sweet potato ..	*Empoasca* sp.	Green jassid
	Sphingidae, unidentified larval form only	Hawk moth
Tobacco ..	*G. operculella*	Potato moth
	H. armigera	Corn earworm
	Thrips tabaci Lind. ..	Tobacco thrips
	Heliothrips haemorrhoidalis Bouche .	Greenhouse thrips
	Empoasca sp.	Green jassid
	O. argentata	Brown jassid
Tomato ..	*H. armigera* .. .	Corn earworm
	Coccidae, unidentified	Scale insect

TABLE 2.—INSECTS RECORDED ATTACKING CROPS ON ARMY FARMS, ADELAIDE RIVER, NORTHERN TERRITORY—JULY, 1945.

Crop Attacked	Insect Pest	
	Scientific Name	Common Name
Cabbage ..	*H. undalis*	Centre grub
	H armigera	Corn earworm
	P. maculipennis	Cabbage moth
Cucumber ..	*C. hilaris*	Pumpkin beetle
	R. palmerstoni	Pumpkin beetle
Citrus	*Aonidiella aurantii* Mask ..	Red scale
	Saissetia oleae Bern. .	Brown scale
	Lepidosaphes beckii (Newm) ..	Mussel scale
Papaw	*Heterodera marioni* (Cornu). ..	Root knot eelworm
Tomato ..	*H. armigera*	Corn earworm
	H. marioni	Root knot eelworm

TABLE 3.—INSECTS RECORDED ATTACKING CROPS GROWN ON ARMY
FARMS AT COOMALLIE CREEK—JULY, 1945.

Crop Attacked.	Insect Pest.	
	Scientific Name.	Common Name.
Beetroot ..	*H. undalis*	Centre grub
	H. recurvalis	Beet webworm
Cucumbers ..	*C. hilaris*	Pumpkin beetle
	R. palmerstoni	Pumpkin beetle
	O. argentata (Evans)	Brown jassid
	Aphis gossypii Glover	Melon aphis
	T. tabaci	Tobacco thrips
	Haplothrips sp.	Black thrips
Tomato ..	*H. armigera*	Corn earworm
	T. tabaci	Tobacco thrips

Twenty-two insect pests and one nematode were identified attacking crops grown on the Army farms at the time of the survey. The most important insect pests at this time were corn earworm on tomato crops, and centre grub and cabbage moth on cabbage crops. A very careful search was made for aphids, and, although the hosts of many species were grown on the farms, only two species were found, the corn aphis on grain sorghum and sweet corn, and melon aphis on curcurbits.

In addition to the insects listed from Katherine River (Table 1) the immature forms of a Pentatomid bug, of a Tingid bug observed feeding on the cotton bolls, and adults of a Pyrrhocorid, *Astemma cingulatus* (Fabr.), were collected from cotton plants. An unidentified Tanypezid fly was also reared from decaying bananas. One tomato crop at a Katherine River farm was seriously affected by corn earworm which migrated from a crop of sweet corn grown on an adjacent field during the " wet " season, and harvested after the tomatoes had been transplanted. Sweet potato crops, in the same area, were seriously attacked late in the season by hawk moth and green jassids. Although the brown jassid, which transmits tobacco yellow dwarf virus disease in southern States, was present, this virus disease was not seen in tobacco crops at the time of the survey.

At Adelaide River cabbage centre grub, corn earworm, and cabbage moth heavily infested a crop of cabbages, which was saved by treatment with 2 per cent. DDT dust in July, followed by a 0·1 per cent. DDT solvent naphtha emulsion applied by a power sprayer in August. Two species of pumpkin beetles were particularly troublesome on cucumbers and melons, and their control with arsenate of lead dust was difficult. Beet webworm was a serious pest of seedling beetroot, in many cases killing the young plants. The green tree ant, *Oecophylla virescens* Fabr., while perhaps not a pest in the usual sense of the word, did select citrus trees at Adelaide River for its

nests, and the black passion bug, *Leptoglossus bidentatus* Montr., attacked a species of *Abutilon* which was being grown on wind breaks. The presence of root-knot eelworm in the seed-beds in this area was so serious that tomato seedlings were obtained from Katherine River for transplanting in the field. Papaws also suffered severely from this pest, and the plantations were very short lived and yielded poorly.

Cucumber and melon crops at Coomallie Creek, like those at Adelaide River, were seriously affected by two species of cucumber beetle, and beet webworm killed many seedling beetroot plants. Only one tomato plant infected with root-knot eelworm was found in this area. Cabbage crops were affected by centre grub and corn earworm, but the severity of attack was not as bad as at Adelaide River.

3. Insecticide Experiments

The profuse and rapid growth of vegetables under warm, tropical conditions, and the large areas involved in the Northern Territory, rendered adequate control of insect pests difficult with the usual types of insecticides. By 1944 it was apparent that there was an urgent need for some rapid mechanical means of applying insecticides to large areas and that more effective insecticides were desirable.

A power row-crop sprayer had already been developed in Canberra. This sprayer consisted of an ordinary type of power orchard sprayer with a 100-gallon vat, and was adapted to take a six-row crop spray boom to which eighteen cyclone, hollow-cone nozzles were attached in such a way that there were three nozzles to each row of plants. Two of the nozzles sprayed horizontally and slightly upwards, and the third nozzle sprayed vertically downwards on to the top of the crop. In this way a very efficient cover of the plants with the spray material was obtained. Two orchard type spray plants were available in the Northern Territory, and these were adapted in this way and were used to treat the tomato and cabbage crops on the farms in 1945. Power dusting machines could have been obtained for use on the farms but this was unnecessary because of the success of the power row-crop sprayers. Such dusts as were necessary, and all dusts in the experiments, were applied by means of a knapsack duster.

Tests had already shown that DDT was very effective against corn earworm, centre grub, and cabbage moth (Helson and Greaves, 1945). Other tests had suggested that synthetic cryolite might be useful should lead arsenate supplies become scarce, and small quantities of crude hexachlorocyclohexane (666), were also available for testing. All these materials were included in the field trials in the Northern Territory in 1945.

(i) *Trials at Katherine River.*

The trials were made at the 1st Australian Experimental Farm at Katherine on a block of 720 cabbages, and a block of 240 tomatoes planted for the purpose. The cabbages were used to test the usefulness of the insecticides against cabbage moth, corn earworm, and centre grub, and the tomatoes were used to test their efficiency against corn earworm.

In the cabbage trial the area was divided into six randomized blocks each containing six plots, with twenty plants per plot. The plants were transplanted on 23rd July, 1945, and were spaced at 2-foot intervals within the rows with 4 feet between each row. Six treatments were included in the trial:—

    1 and 2 per cent. 666 " pyrophyllite " dusts;

    0·5 and 1 per cent. DDT " pyrophyllite " dusts;

    40 per cent. synthetic cryolite with " pyrophyllite " dust;

    control.

These were applied with a knapsack duster at the rate of approximately 35 lb. to the acre. The first application was made on 14th August, and three subsequent applications followed at 10-day intervals.

At the end of September none of the plants was injured by corn earworm, but cabbage cēntre grub attack had begun. Accordingly each plant in the plots was rated for centre grub damage on a five-point scale, as described by Greaves (1945). These ratings are given in Table 4.

TABLE 4.—RATING FOR DAMAGE BY *H. undalis* TO CABBAGES AT KATHERINE, NORTHERN TERRITORY, AND MEAN RATING FOR DAMAGE PER TREATMENT, 30TH SEPTEMBER, 1945.

Treatment

1 Per cent. 666.	2 Per cent. 666.	40 Per cent. Cryolite.	1 Per cent. DDT.	0 5 Per cent DDT	Control.
3	0	1	0	0	31
0	0	0	13	0	38
0	0	0	0	0	21
0	0	0	0	0	33
0	0	0	0	0	22
12	24	0	0	0	29
Total 15	24	1	13	0	174
Mean rating 2·5	4·0	0·16	2·16	0	29

The sudden cessation of hostilities, and the changes in Army personnel subsequently following, prevented the experiment being carried through to harvest of the cabbage heads. However, it will be seen that all of the treatments used were effective against cabbage centre grub, that there was a positional effect at the end of the blocks in the 666 treatments, and that there was no significant difference between the treatments.

The block of 240 tomato plants in the corn earworm trial was divided into eight randomized sub-blocks, with six plots per sub-block and four plants per plot. The plants were transplanted on 17th July,

1945, and were spaced 6 feet apart within the rows with 8 feet between rows and 10 feet between each plot. The treatments were—

> 0·1 and 0·2 per cent. DDT solvent naphtha spray;
> 1 and 2 per cent. DDT " pyrophyllite " dusts;
> 50 per cent. synthetic cryolite " pyrophyllite " dust;
> control.

The dusts were applied at approximately 35 lb. to the acre, and the sprays at approximately 80-100 gallons to the acre. The first application was made on 31st August, followed by three subsequent treatments at 10-day intervals.

Two harvests of fruit were made, the first at the end of October, and the second about fourteen days later. At harvest, a record was kept of the number of fruits infested with corn earworm and the number of sound fruit. The figures obtained are set out in Tables 5 and 6.

TABLE 5.—NUMBER OF TOMATOES INFESTED WITH *H. armigera* PER REPLICATE AT FIRST HARVEST ON 3RD OCTOBER, 1945, LEFT-HAND FIGURES, COMPARED WITH THE NUMBER OF SOUND FRUIT, RIGHT-HAND FIGURES. THE MEAN PERCENTAGE INFESTATION IS SHOWN IN THE LAST LINE OF THE TABLE.

Block Number	Treatment.					
	0·2 Per cent. DDT	0·1 Per cent. DDT	2 Per cent DDT dust.	1 Per cent. DDT dust.	50 Per cent. Cryolite.	Control
1	1/40	0/48	5/65	0/36	0/43	20/66
2	0/48	0/58	0/52	0/67	0/47	3/46
3	0/39	0/49	0/50	0/30	0/50	6/71
4	1/33	0/30	0/42	0/34	0/32	8/41
5	0/44	1/57	0/34	0/38	0/35	5/37
6	0/30	0/52	0/50	0/37	0/35	1/27
7	0/40	0/49	0/64	0/61	0/43	6/51
8	0/51	0/41	0/40	3/44	0/47	5/57
Mean percentage infested ..	0·6	0·3	1·3	0·9	0·0	13·6

TABLE 6.—NUMBER OF TOMATOES INFESTED WITH *H. armigera* AT SECOND HARVEST, MIDDLE OF NOVEMBER, 1945, LEFT-HAND FIGURES, COMPARED WITH THE NUMBER OF SOUND FRUIT, RIGHT-HAND FIGURES. THE MEAN PERCENTAGE INFESTATION IS SHOWN IN THE LAST LINE OF THE TABLE.

Block Number.	Treatment					
	0·2 Per cent. DDT	0·1 Per cent. DDT	2 Per cent. DDT dust.	1 Per cent. DDT dust.	50 Per cent. Cryolite dust.	Control.
1	0/50	5/59	5/60	0/80	0/40	10/50
2	0/47	0/60	0/48	0/46	0/53	5/60
3	0/41	0/47	0/48	0/51	0/61	10/73
4	0/46	0/34	1/43	0/47	1/41	9/44
5	0/39	7/57	0/38	0/30	2/43	2/24
6	0/21	0/54	0/67	2/15	0/55	3/40
7	0/54	0/56	0/48	0/42	0/51	5/57
8	0/46	0/54	0/47	0/47	0/51	9/71
Mean percentage infested ..	0·0	1·2	1·5	0·6	0·8	12·6

Analysis of these data is not necessary. The controls are significantly different from all other treatments, and the damage on each of the treated plots is so small that it is not possible to discriminate between the treatments.

(ii) *Adèlaide River.*

At Adelaide River a small experiment with tomato plants was carried out to test the usefulness of a mixture of dichloropropane and dichloropropylene* as a fumigant against root-knot nematode, *Heterodera marioni.* In July, 160 tomato plants were transplanted in soil which had been infested with eelworm the season before. The plants were set out on a 6 × 3 Latin square design with eighteen plots each 12 × 24 ft. There were nine plants to each plot, planted 4 feet apart within the rows and 8 feet between each row. The fumigation of the plots was carried out ten days before sowing the seed, using a hand injector to apply the fumigant to a depth of 6 inches at the rate of 200 lb. to the acre. There were three treatments—

   (1) Fumigation of seedbed and plot;
   (2) fumigation of seedbed only and transplanting into an untreated plot;
   (3) no fumigation of either seedbed or plot.

The sudden cessation of hostilities immediately affected the experiment, which also could not be carried to completion. However, in September every plant was lifted and its roots examined and rated for eelworm by the method described by Stark *et al.* (1944). At this time it was too early to obtain satisfactory results, but the observations showed that there was less eelworm attack on the roots of plants taken from the fumigated seedbed and plot and the fumigated seedbed only, suggesting that the fumigant was having a beneficial effect.

4. Acknowledgment

· 5. References

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* D–D.

Investigations on the Control of Oriental Peach Moth, *Cydia molesta* Busck., in the Goulburn Valley, Victoria

*By G. A. H. Helson, M.Sc.**

Summary.

Six species of parasites of the Oriental peach moth, *Cydia molesta* Busck., were introduced into the Goulburn Valley, Victoria, from the United States of America between 1935 and 1939, and 14,195 parasites of all species were released up to 1940, after which there were no further liberations. Three of the species released were recovered from field collections, but two of these have not been recovered since 1938 and it must be presumed that these two have failed to become established. There were 964 recoveries made of the other species, *Macrocentrus ancylivorus* Rohr., over a widely dispersed area during the period 1935 to 1940, but only four of these were from overwintering parasites. The number of recoveries decreased sharply after liberations ceased, and as no further recoveries were made after the 1939-40 season, it is unlikely that this species has succeeded in establishing itself either. Laboratory observations over a number of years suggest that this failure may be due to the long, warm autumns and lack of alternative hosts on which the parasite might overwinter.

Recent field experiments show that useful control of *C. molesta* can be achieved in the fruit of late canning peaches by two applications of a spray containing 0·1 per cent. DDT, the first applied six weeks and the second three weeks before harvest.

Earlier reports on the Oriental peach moth, *Cydia molesta* Busck., in the Goulburn Valley, Victoria, have been published by Gay (1935) and Helson (1939). Up to the time of the second report no satisfactory method of chemical control had been found, and thereafter, the emphasis was placed on biological control. The work was discontinued in 1941 because of the greater urgency of other horticultural problems associated with the war. It reopened in 1945, when sufficient DDT became available for a small field trial, and was continued in 1946.

The present paper gives details of the introduction, liberation, and recovery of parasites, for the most part since 1938, and the results of recent field trials with DDT.

1. Attempts at Biological Control

(i) *Introduction of the Parasites.*

Field recoveries of *Macrocentrus ancylivorus* Rohr., up to the end of the 1937-38 season, had suggested that this was the most promising of five introduced parasites (Helson, 1939), and that it was capable of becoming established during the summer and autumn months. It was not known, at this time, whether the insect could overwinter in the Goulburn Valley. Additional introductions of this parasite were made from New Jersey in the spring of 1938-39, and again in 1939-40, together with three others, *Macrocentrus delicatus* Cress., introduced for the first time from Southern Ohio, *Inareolata molestae* Uch., which had been introduced into New Jersey from Japan and Korea, and *Glypta rufiscutellaris* Cress. The material was received in four shipments, two in each season. The numbers of peach moth cocoons exposed to parasites in America and received in Australia in these shipments, together with the number of parasites which emerged, are presented in Table 1.

* An officer of the Division of Economic Entomology.

TABLE 1.—NUMBER OF ORIENTAL PEACH MOTH COCOONS EXPOSED TO PARASITES AND RECEIVED FROM THE U.S.A., AND THE NUMBER OF PARASITES WHICH EMERGED FROM THEM.

Number of Shipment	Date of Receipt.	Total Number Moth Cocoons	Number of Parasites which Emerged			
			M.ancylivorus	M delicatus.	I molestae	G.rufis-cutellaris
5	23rd Sept., 1938	21,448	1,076	196	113	..
6	28th Nov., 1938	20,051	3,224	191	..	..
7	28th Nov., 1939	20,000	1,707	..	..	..
8	22nd Nov., 1939	40,000	3,387	..	..	34
Total number of parasites which emerged			9,394	387	113	34

Shipments 6 and 7 were delayed in transit, taking 67 and 97 days respectively to arrive from New York, as against the average of 50 days. Nevertheless, all arrived in good condition, and in time to enable liberations to coincide with a maximum abundance of the moth larvae in the field. Thus the parasites had every opportunity of becoming established.

' Table 2 shows the total number of parasites of all species which have been liberated since 1935. These liberations were made in badly infested orchards throughout the Shepparton and Ardmona districts (Fig. 1).

The material of *Bassus diversus* Muesb. liberated in 1939 was derived from a laboratory stock, the progeny of earlier introductions.

TABLE 2.—TOTAL NUMBER OF PARASITES OF ALL SPECIES LIBERATED IN THE GOULBURN VALLEY, VICTORIA, FROM 1935 TO 1940.

Species	Season of Liberation.	Localities	Number Liberated			Total Number of Each Species
			Male	Female	Total	
Macrocentrus ancylivorus Rohr.	1935–36	1 and 2 ..	25	37	62	..
	1936–37	2, 7, 8, and 9	108	128	236	.
	1937–38	1, 2, 5, 6, 7, and 13	1,435	1,489	2,924	.
	1938–39	13.. ..	2,024	1,388	3,412	.
	1939–40	1, 2, 3, 5, 6, 7, 8, 10, 11, 13, 14, 15, 16, 17, 18, and 19*	2,606	2,446	5,052	..
	1940–41	Bamawm Extens on	20	20	40	11,726
Glypta rufiscutellaris Cress.	1935–36	1	15	26	41	..
	1936–37	3, 8, and 12	175	203	378	.
	1937–38	3, 4, 6, and 10	332	398	730	1,149
Bassus diversus Muesb. ..	1937–38	6 .. .	7	791	798	.
	1938–39	5 and 12 ..	..	45	45	.
	1939–40	13.. ..	..	102	102	945
Inareolata molestae Uch. ..	1937–38	4 and 6 ..	9	33	42	.
	1939–40	13.. ..	13	23	36	78
Ascogaster carpocapsae Vier.	1937–38	6	..	..	164	164
M. delicatus Cress. ..	1938—39	13.. ..	69	64	133	133
Total number of all species						14,195

*Locality 19 is not shown in Fig. 1; it is situated at Kyabram, approximately 20 miles north-west from the nearest point of liberation in Ardmona.
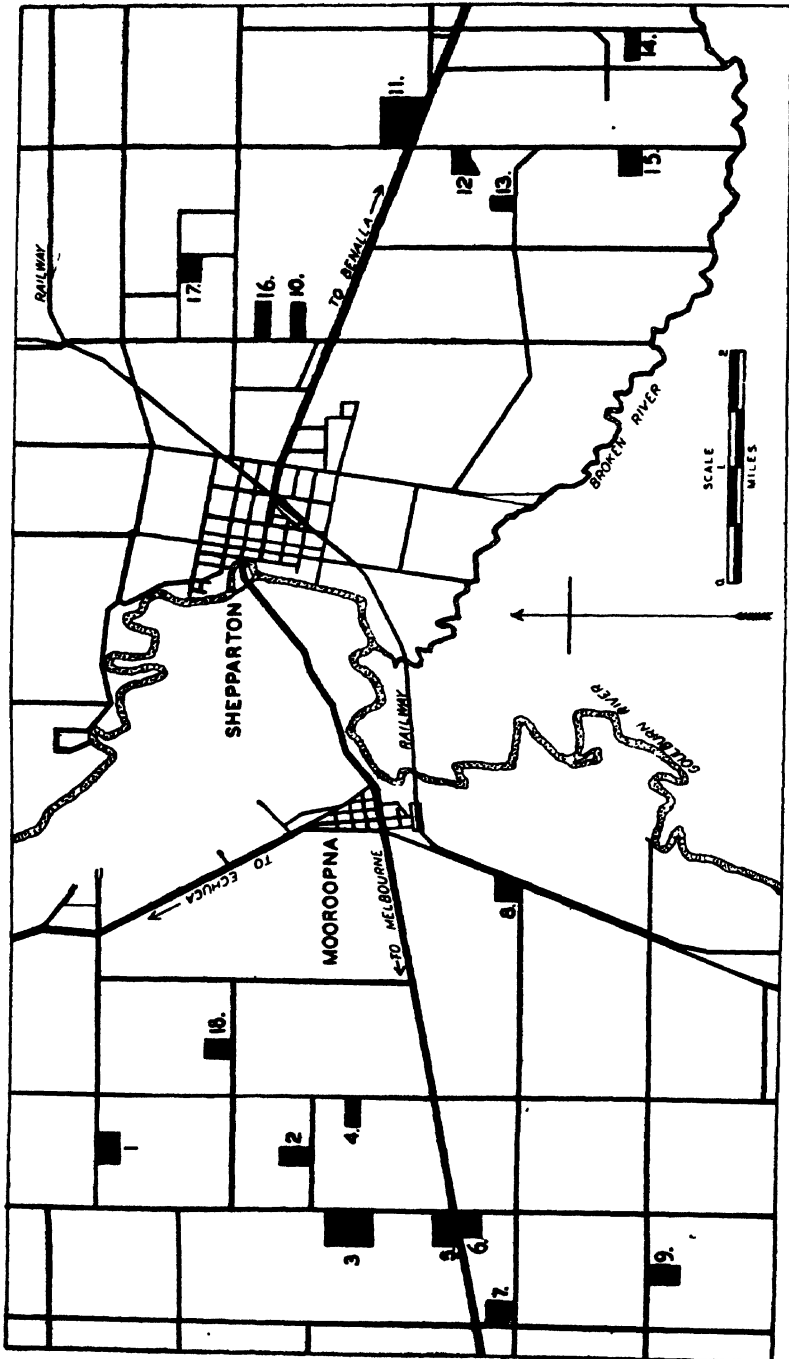
F<small>IG</small>. 1.—Map of Ardmona and Shepparton districts showing the orchards where parasites have been liberated since 1935.

(ii) *Recovery of the Parasites.*

Three of the five species of parasites released prior to 1938, *M. ancylivorus, B. diversus,* and *G. rufiscutellaris,* were recovered from field collections made from 1936 to 1938 (Helson, 1939), but since then no further recoveries have been made of the latter two species, notwithstanding the further liberation of *B. diversus* in 1939. No recoveries of *Ascogaster carpocapsae* Vier., *M. delicatus,* or *I. molestae* have been made at any time.

Additional recoveries of *M. ancylivorus* were made in the spring of 1938-39, when four parasites emerged from overwintering cocoons collected at localities 5 and 6 the previous autumn and stored in the laboratory during the winter. More were recovered later from peach moth larvae of the same season (Table 3). The recoveries were much lower than in the previous season, and were not made until after further liberations in December. The low numbers may be accounted for by the lack of twig infestation and the extremely hot weather in January and February.

TABLE 3.—NUMBER OF *M. ancylivorus* RECOVERED FROM LIBERATION SITES IN 1938-39.

Date Collected.	Locality.	Number of Moth Larvae Collected.	Number of Parasites Recovered
October, 1938	5	12	..
	6	30	..
November, 1938	3	298	..
	6	20	..
	8	72	..
December, 1938	3	284	..
	5	10	..
	6	300	..
	7	87	..
	13	34	..
January, 1939	3	428	2
	5	4	..
	6	216	..
	7	113	1
	8	7	1
	13	236	..
February, 1939	3	241	3
	6	241	3
	7	30	..
	13	41	..
March, 1939	6	10	..
	13	12	..
Total	..	2,726	10

In 1939-40 no *M. ancylivorus* were bred from overwintering larvae collected late the preceding autumn, but a laboratory colony of the parasites overwintered, although the mortality was high. Table 4 summarizes the recovery of parasites later in the season. Greater

numbers of *M. ancylivorus* were recovered than in the preceding season, and in addition small numbers of an *Inareolata* distinct from *I. molestae* appeared in the collections. This species is either a native, or was introduced accidentally prior to the commencement of the present investigation. The seven *M. ancylivorus* (all males) recovered from locality 3 in December were bred from infested twigs collected the previous month. They emerged twelve months after the last liberation (of 115 females) had been made at this point in December 1938, and probably belonged to the fourth generation bred in the field. This recovery shows that at least some of the individuals of this species withstood the heat waves of the preceding summer, and then overwintered successfully in the field on Oriental peach moth.

TABLE 4.—NUMBER OF PARASITES RECOVERED FROM LIBERATION SITES IN 1939-40.

Date Emerged.	Locality.	Number of Moth Larvae. Collected	M ancylivorus.	Inareolata sp.
December, 1939	3	713	7	15
	6	289	..	3
	10	140	..	1
	13	97	..	1
January, 1940	3	336	11	2
	6	104	12	1
	7	62	..	.
	8	7	..	.
	10	177	..	1
	12	13	..	..
	13	55	3	5
February–March, 1940	3	328	16	3
	6	371	53	4
	7	93	1	1
	8	24	1	1
	10	178	15	2
	13	61	27	2
	16	54	9	..
Total		3 109	155	42

No parasites emerged in the spring of 1940-41 from cocoons collected at the end of the previous season, and of 200 overwintering cocoons of laboratory-bred *M. ancylivorus* only 54 emerged. During the remainder of the 1940-41 season 3,828 moth larvae, collected from infested tips at all liberation sites, yielded only one *M. ancylivorus* from a collection made at locality 6 in October 1940. This was the last recovery of an introduced parasite, although field surveys were made each December for the next three' years in the districts where releases had been made.

(iii) *Discussion.*

Thus a total of 14,195 parasites, including 11,726 *M. ancylivorus* was liberated since 1935; and 964 were recovered. However, it is clear from the tables that almost all the recoveries closely followed

the releases. Only four parasites were bred from overwintering cocoons, although seven field recoveries of first generation *M. ancylivorus* in 1939, twelve months after the last liberation at this site, were undoubtedly the progeny of small numbers of this species which had overwintered and begun breeding in the spring. As there has been no evidence of the presence of any introduced parasite since 1941, it is unlikely that any of them succeeded in establishing themselves.

The percentage infestation of peaches brought to canning factories during the period covered by the liberations ranged from 4 to 50 per cent., and during the years of heaviest liberations stood at about 20 per cent., so that lack of host larvae could hardly have contributed to the failure of the parasites to establish themselves. On the other hand, three years' observations revealed that the long warm autumns caused a high percentage of *M. ancylivorus* to emerge in May and June each year, when no hosts were available. Thus there was a high mortality of parasites that ought to have overwintered. There was also a high mortality in overwintering parasites. In addition there was an absence of such alternative hosts as *Ancylis comptana* Froehl., which is present in the United States, the larvae of which provide hosts in the autumn and greatly assist *M. ancylivorus* to overwinter.

2. Chemical Control

Of 44 insecticides previously tested in the laboratory, the only one which had shown any promise was a mixture of white oil, nicotine sulphate, and bentonite (Helson, 1939). However, as the field tests with this mixture gave an unsatisfactory control, the investigation was suspended at the end of the 1937-38 season. In 1944-45, when a quantity of DDT was made available for experimental purposes, a small field test was planned to determine whether this material was of any value in the control of Oriental peach moth.

The experiment was made in Ardmona, Victoria, on a block of 169 young Pullar Cling peaches (a late canning variety), which were just coming into bearing and were subject to moderate tip infestation by Oriental peach moth. Thirty-six trees were selected on a randomized block design for two treatments, sprayed and unsprayed, each experimental tree being surrounded by buffer trees. Two applications of 0·1 per cent. DDT-solvent naphtha emulsion (Helson and Greaves, 1945) were given in 1945, and "Rucide," a commercial water-soluble DDT preparation, was used at the same concentration in 1946. The applications were made late in the season with the object of protecting the fruit from attack, the first on February 6 in both 1945 and 1946, and the second on February 21 in 1945 and on February 28 in 1946. Approximately two gallons per tree were applied in 1945 and three gallons per tree in 1946, at a pressure of 350 lb. per square inch. In 1946 heavy autumn winds caused some of the fruit to drop, but all such fruit was examined for infestation and the figures included in the record.

In the 1945 experiment, the fruit was harvested on March 3. Details of this harvest are presented in Table 5. The difference between treatments is significant at the 0·1 per cent. level.

TABLE 5.—RESULTS OF SPRAY TRIAL USING 0·1 PER CENT. DDT–SOLVENT NAPHTHA EMULSION FOR THE CONTROL OF ORIENTAL PEACH MOTH IN ARDMONA, VICTORIA, 1945.

-	Treated Trees.	Untreated Trees
Number of sound fruit	1,949	1,659
Number of moth-infested fruit	73	116
Total number of fruit	2,022	1,775
Percentage infestation	3 6	6 5

It will be seen that the two treatments of 0·1 per cent. DDT protected the fruit to an appreciable extent. Analysis of the fruit at harvest showed that there was a DDT residue of approximately 9 p.p.m.* A portion of the sprayed fruit was canned in the normal way, and an analysis of the syrup and fruit after canning failed to show any trace of DDT. Thus two applications of 0·1 per cent. DDT., the first six weeks and the second three weeks before harvest, did not produce a DDT residue in excess of that permitted by Health Regulations (10 p.p.m.), and no DDT was carried through the canning process into the manufactured product.

The harvest in 1946 was on March 26. The results are shown in Table 6. The difference between treatments is significant at the 0·1 per cent. level.

TABLE 6.—RESULTS OF SPRAY TRIALS USING 0·1 PER CENT. DDT IN THE FORM OF " RUCIDE " FOR THE CONTROL OF ORIENTAL PEACH MOTH IN ARDMONA, VICTORIA, 1946.

-	Treated Trees	Untreated Trees
Number of sound fruit	2,752	2,644
Number of moth-infested fruit	78	262
Total number of fruit	2,830	2,906
Percentage infestation	2·75	9·0

Infestation of the fruit was again light, and showed a slight increase over the preceding season, but again useful reduction of infestation was obtained by the application of two late sprays to the fruit.

* The DDT analyses were made by Mr. R. F. Powning of the Division of Economic Entomology.

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The Use of DDT for the Control of the Buffalo Fly (*Siphona exigua* (de Meijere))

By K. R. Norris, M.Sc.*

Summary.

Experiments on the protection of cattle against the buffalo fly (*Siphona exigua* (de Meijere)) by the use of preparations containing DDT are described. The spraying of only half the cattle in a herd results in the virtual elimination of flies from the untreated beasts. It is unnecessary to spray the entire body surface of the animals to bring about control of the flies, spraying on a patch over the shoulders being sufficient. When all the cattle in a herd are thoroughly sprayed over the shoulders with emulsions containing 4 per cent. DDT, or with DDT suspensions prepared from 1 per cent. Rucide (see page 28) they remain free of buffalo flies for approximately two weeks, after which there is a gradual increase in the fly population, usually necessitating spraying again at three weeks. As this period generally coincides with the intervals between dippings for cattle tick control, the two treatments may be applied in the course of the same muster.

Milking cows may be kept relatively free of buffalo flies by lightly spraying them with kerosene solutions of DDT. To avoid skin injury such sprays are applied as a fine mist from an ordinary household "Flit gun," which leaves a small, superficial deposit of DDT of low persistency. However, the daily handling of milkers permits treatment as often as necessary.

Large numbers of cattle, dipped or sprayed over the entire body surface with DDT preparations for the control of the cattle tick remained virtually free of buffalo flies for at least five weeks. It is suggested that the treatment resulted in the complete elimination of the fly from the large area on which the cattle grazed, so that reinfestation could only occur by the infiltration of flies from the surrounding areas. On small properties reinfestation is generally rapid, even if the protective period of the spray outlasts the life cycle of the fly.

Some factors influencing the persistency of DDT on cattle are discussed. Up to a point, persistency of emulsions and solutions varies with the concentration of DDT in the spray. The loss of DDT faster from cattle than from inanimate surfaces is considered to be due largely to rubbing and licking by the beasts, whilst the direct influence of rain and insolation in the removal of DDT is not considered of prime importance.

1. Introduction

In a previous paper (Norris, 1946) the testing of traps for the control of the buffalo fly on dairy cattle was described. The present publication is an account of experiments on the control of the fly on dairy and beef cattle by the use of DDT preparations. This material proved to have such outstanding qualities for use against the buffalo fly that attention was concentrated on its testing to the virtual exclusion of all other insecticides. The important attributes of DDT are that a buffalo fly alighting on a deposit is killed by the small quantity that it picks up on its appendages, and that sufficient quantities persist for considerable periods on treated surfaces to kill buffalo flies alighting there. The spraying of cattle with DDT preparations therefore has unique advantages for the control of the buffalo fly, which, in areas devoid of the water buffalo, is practically specific to

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* An officer of the Division of Economic Entomology.

cattle, the flies spending almost their entire lifetime on the skin of the host. Although it has been found that persistency on living cattle is not as great as on inanimate surfaces, DDT is so effective against the buffalo fly that its use is now freely advocated as a practical and economical method of controlling this pest. The work described in this paper was carried out principally at Malanda, North Queensland. Notes on certain aspects of it have already been published by Legg (1945), Belschner (1946), and Roberts (1946).

2. Methods of Measuring Persistency of DDT on Cattle

The following are some possible methods of investigating the persistency of deposits of DDT on cattle:

(i) *Chemical Analysis of Extracts from Hair Clippings.*

The disadvantage of this method is that samples must be large to permit an accurate analysis for p,p'–DDT, particularly when only low concentrations are present. Both this and the succeeding method (ii) are useful for the comparison of the persistency of various preparations.

(ii) *Bioassay Tests of Clipped Hair Samples, Using the Buffalo Fly as the Test Insect.*

Quite small samples are sufficient for the performance of such tests, and testing is a relatively simple matter. The method adopted is as follows:

The samples of hair are spread out under petri dishes on paper squares glued to a wooden baseboard. The baseboard and paper are perforated by 1-inch holes directly below each petri dish. Flies are netted from untreated cattle, and, taking advantage of their strong attraction to light, transferred as soon as possible after capture into the petri dishes from beneath. The holes in the baseboard are then plugged with clean cotton wool, and the boards carrying the petri dishes placed under a strong light to activate the flies. DDT does not kill flies immediately on contact. The earliest detectable symptom is restlessness, and "excited" behaviour. A period of increasing incoordination of the legs follows, until finally the fly is unable to stand, but lies kicking or buzzing on its back or side. After the loss of the power of locomotion, the vigour of the movement of the appendages gradually decreases, and the final trace of life is the twitching of the terminal segments of the tarsi. The greater the concentration of the DDT on the surface on which the fly crawls, the more rapidly do these symptoms succeed one another, so that, by assigning ratings to the different recognizable stages of the paralysis (unaffected, slightly affected, strongly affected but still able to walk, unable to walk but still vigorously moving, almost immobile, and apparently dead), a comparison may be made of the relative concentration of DDT on different samples.

Disadvantages of this method are that it is not possible to obtain a "standard" buffalo fly comparable with the "standard" housefly of the Peet-Grady test, and that under conditions prevailing on a field station, experiments cannot be carried out under conditions of constant temperature and humidity; but in spite of these drawbacks useful information may be obtained.

(iii) *Insectary Tests*.

The treated beast may be enclosed in a gauze insectary, and observations made on the survival of buffalo flies liberated inside the enclosure. Disadvantages of this method are the small number of beasts that may be handled in any one experiment, and the strong possibility that the insectary may become contaminated with DDT through the contact of cattle with the rails, and the shedding of DDT on to the floor. Also buffalo flies sometimes tend to leave a beast in an insectary, and rest on the gauze.

(iv) *Study of the Trend of the Fly Population on a Herd Following Treatment with DDT*.

DDT is not a repellent, and the rate at which it affects flies varies as the concentration on the surface on which the flies alight. When a herd of cattle is treated with a DDT preparation, all the flies present at the time of spraying are eliminated. Those flies (emerging from pupae in the pasture, or migrating from cattle outside of it) which first attempt to reinfest the treated cattle are affected by DDT so rapidly that they are " knocked down " soon after alighting on the hides. Thus the infestation visible at any time shortly after spraying is practically nil. As the concentration of the DDT on the hides is reduced, the flies reinfesting the cattle are able to survive for longer periods, and so a gradual increase in population results, even while some DDT remains on the hides. Some of the important factors affecting the rate of reinfestation of cattle after they have been freed of flies are as follows:

(*a*) Natural variation of the buffalo fly population with seasonal conditions and biological factors. On the Atherton Tableland the fly population is at its peak in January and February, when reinfestation is rapid once a DDT spray has worn off. On the other hand, in July and August, when the fly population is at a low level, reinfestation is very slow, even if temperatures are temporarily favourable.

(*b*) Direct effect of weather on the activity of the flies, and hence on the rate of infiltration from untreated to treated areas. Cool conditions slow up the activity of the fly, and hence tend to retard reinfestation, even if not immediately reducing the numbers of the flies.

(*c*) Size of property on which the treated stock graze. The average area of dairies on the Atherton Tableland is only about 150 acres, and so very rapid reinfestation is likely to occur if surrounding properties carry untreated cattle. On the other hand a cattle station many square miles in area would be subject to only slow reinfestation from outside the boundaries.

As method (iv) provides information directly in terms of fly population, as is required from the point of view of the practical problem, it has been most employed, though supplemented at times by other tests, particularly bioassays.

3. Effect on the Population of *Siphona exigua* of Treating Cattle with DDT Preparations

DDT has been found to be highly effective against the cattle tick (*Boophilus australis*) which is an important pest throughout most of the area of distribution of the buffalo fly in Queensland. The possibility of DDT coming into general use for cattle tick control has an important bearing on the buffalo fly problem. As will be described below, it is unnecessary to spray the entire body surface of a herd of cattle to control the buffalo fly. On the other hand, complete coverage of the body of a beast is necessary for tick control, so that if DDT be correctly employed against the tick, the buffalo fly population is automatically eliminated. Use of DDT primarily for the control of the buffalo fly therefore involves different treatment from its employment primarily for the control of the tick. The two methods will be dealt with separately.

(i) *Use of DDT Primarily for Buffalo Fly Control.*

In spraying for buffalo fly control, two methods may be adopted. The hides of the cattle may be saturated with sprays of DDT, leaving a deposit of considerable persistency which will afford lasting protection against the fly, or else they may be sprayed fairly frequently with a light-mist spray of low persistency. Both methods have their uses, though the former is by far the more widely applicable.

(a) *Use of Saturating Sprays to Obtain Prolonged Persistency.*

As will be shown in a later section, persistency of DDT on cattle is dependent partly on the initial dose. Thus a heavy spraying, saturating the hair right to the skin surface, is the most effective way of applying any one type of spray. For use in this manner, sprays must be harmless to the skin of cattle. In this research early experiments were carried out with potentially harmful kerosene sprays, and are described hereunder merely for purposes of illustration, without suggesting that the sprays should be used nowadays for practical purposes in the manner described.

(1) *Preparations employed.*—The first experiments were carried out with straight solutions of DDT, principally in kerosene. As soon as suitable emulsions had been devised, the use of solutions was discontinued, because of the likelihood of injury to the cattle. Desquamation, particularly of the neck, was noted on a number of cattle sprayed with kerosene solution, and one herd showed evidence of loss of control of body temperature, as described by Freeborn *et al.* (1934). Emulsions tested were only those likely to be produced in quantity in Australia, the most important being power kerosene emulsion, solvent naphtha 90/190 emulsion, and *Eucalyptus dives* type oil emulsion, using "Wetsit" as the emulsifying agent (see Hackman, 1946). All proved satisfactory, and there were no indications of injury to the skin of cattle from their use. No outstanding differences in persistency between the different solutions and emulsions have been noted.

Towards the end of the phase of the research with which this paper deals, Messrs. Taubmans' "Rucide" was made available for study. This is a paste containing 50 per cent. by weight of p,p'–DDT and also a "solubilizer." The paste is melted until a clear liquid is formed, at a temperature in excess of 100°C. The melt is then poured

into soft or softened water, which is vigorously stirred during the pouring, a suspension of fine particles of DDT resulting. This preparation has considerable stability, and is apparently completely harmless to the skin of cattle.

(2) *Equipment employed.*—Large, continuous-spraying, hand atomizers were employed, also knapsack sprays to which several yards of hose had been fitted to enable one man to operate the nozzle, while another carried and operated the reservoir. For the small user such appliances are probably quite satisfactory, but for large-scale treatment of cattle with the object of controlling the buffalo fly, a motor-driven pump of some type would be desirable.

(3) *Results of experiments.*—The following experiments were carried out on dairy cattle at Malanda, North Queensland, in 1944. Continuous-spray, hand atomizers were employed to apply 4 per cent. DDT in lighting kerosene, so that the hair of the treated area was thoroughly saturated, without run-off. Although this preparation would not now be used in experiments, the results obtained in the control of the fly population serve to illustrate the development of the principles for the use of DDT against the fly.

(i) On February 8, 1944 a herd of 28 cows, 10 calves, and 1 bull were sprayed on the neck, back, and sides. Before treatment the cows bore an average of about 50 flies each. After spraying they remained practically free of flies for 8 days. From the 8th to the 15th day the mean daily infestation averaged less than a dozen flies per beast. After the 15th day the fly population rose beyond the pre-treatment level, and it was presumed that the DDT was no longer effective. The area of this property was 120 acres, and it was bounded on most sides by properties carrying untreated cattle from which the flies could migrate on to the treated cattle, surviving in increasing numbers as the DDT progressively wore off.

Eight days after treatment, 12 untreated, heavily infested calves broke into the paddock where the sprayed calves were kept. When the strange calves were removed from the paddock, considerable numbers of flies were left on the treated calves, but in less than 12 hours they were again free of flies, demonstrating that the DDT was still very active on their hides.

(ii) A herd of 48 milkers, 3 bulls, and 7 calves was treated on February 18, 1944, on the neck, back, and sides of each animal, leaving unsprayed those regions in reach of the tail in the anticipation that any flies alighting there would be driven on to the treated parts. Probably the spraying was done somewhat more heavily than in the case of experiment (i). Before spraying the cows were heavily infested, individuals carrying up to 1,000 flies, the mean being estimated at about 300 flies per beast. Practically no flies were seen on the milkers for 10 days after spraying. Until the 22nd day the highest mean number per cow at any one count' was 10·5, though generally less. After the 22nd day the mean numbers ranged about 25 per beast, and did not regain their pre-spraying level before observations were discontinued on the 35th day after spraying. It is probable that the failure of the fly population to restore itself towards the end of this period was partly due to seasonal conditions. The area of the property was 167 acres, and reinfestation was possible along most of the boundaries.

An interesting comparison is provided by the history of 16 dry cows grazing on a road running past the property, which were also sprayed, but somewhat more lightly than the milkers, on February 18. In the course of their grazing along the road, these cows frequently came in close proximity to numerous untreated cattle. This fact and the lighter spraying may have been responsible for the fact that the dry cows were reinfested much earlier than the milkers. From the 8th day onwards the infestation was more or less comparable with that which reappeared on the milkers after the 22nd day.

(iii) On March 17, 1944, in a herd of 24 cows grazing on an area of 120 acres, 12 were selected as consistently carrying the heaviest infestations (mean 74 before spraying). The mean number of flies on the remaining cows was 32. The 12 most heavily infested cows and the bull (pre-spraying infestation approximately 500) were sprayed on the neck, back, and sides as far back as the hip.

All flies were immediately eliminated from the sprayed beasts. About 15 hours after spraying the mean infestation on the unsprayed milkers had dropped to 4 flies per beast, and there was less than 1 fly per untreated beast 22 hours after spraying. For 11 days after spraying the mean daily average number of flies on the sprayed milkers was less than 1 per beast. The average infestation on the untreated cattle during this period was generally less than 2 per beast (max. 4), though always in excess of the mean on the treated cattle. From the 12th to the 22nd day the numbers remained low, usually not more than 5 per beast, though generally slightly higher on treated than on untreated cattle. After the 22nd day there was a great increase in the mean numbers of flies.

This experiment demonstrated that flies move around from beast to beast frequently enough to bring about their control if only half the cattle in a small herd are treated. Laake (1946) reports similar results from experiments on the control of the horn fly (*S. irritans* (L.)) with DDT preparations. Since the performance of this test, other experiments have amply demonstrated that when cattle treated with DDT mix with untreated cattle, reduction or elimination of the flies on the untreated cattle takes place. In a further experiment the bull and every fourth cow in a herd was treated, but control was not as satisfactory as when half the number of cows was sprayed.

(iv) On April 21, 1944, a herd of 39 cows and a bull, grazing on a property 161 acres in area, were sprayed in patches about 18 inches by 12 inches on either flank, and similar areas on either side of the withers, these being the parts of the hide where the flies tend to congregate in the greatest numbers. Before spraying each cow carried an average infestation estimated at about 150–200 flies, and the bull about 2,000 flies. Until the 12th day after spraying not more than 1 fly per cow was recorded at any observation, although the bull had been observed to carry up to 9 flies during this period. The mean infestation per milker was not recorded as higher than 2 flies per observation between the 12th and the 21st days, by which time it had become doubtful whether adverse weather conditions were not hindering the reinfestation of the herd. Consequently about 400 flies netted from untreated cattle were released on the herd on the morning

of the 22nd day. There was a considerable survival of flies when the cows were brought in for the afternoon milking about 4 or 5 hours after the liberation, so it was concluded that the deposit of DDT had substantially worn off. Chemical analysis of hair samples clipped from the shoulders of cows on the 18th day after treatment showed that there was a considerable amount of DDT on the shoulders at that time (see Table 1). Infestations on untreated cows in the vicinity of the farm averaged about 200 flies per beast on the 12th day, while at 22 days untreated stock on a roadway adjoining the farm had a mean infestation of about 50 flies per head. The evidence suggests that the protection of the shoulder and flank spraying extended into the 3rd week, although the results were somewhat confused by the onset of cool weather conditions after the commencement of the test.

(v) Subsequent to the completion of this series of tests with lighting kerosene solution, numerous experiments were carried out with emulsions and Rucide, in which the shoulders only of all the cattle in a herd were sprayed, resulting in satisfactory control of the fly population. For instance, 30 head of beef cattle were sprayed over the shoulders with a 4 per cent. emulsion of DDT on November 12, 1945, at Innisfail, North Queensland. Before spraying the infestation averaged about 200 flies per beast. A fortnight later the fly population averaged only 6 flies per beast, and bioassay of hair samples showed distinct traces of DDT on the shoulders.

Evidence has been secured which suggests that DDT persists longer on the shoulders than on the flanks (see Table 1 and Section 3 (ii). The significance of such observations is that even if a beast be sprayed over the entire body, the final result, after a preliminary period of differential removal of DDT is much the same as if only the upper parts had been sprayed.

A summary of information of other miscellaneous experiments with saturating sprays of DDT in the form of emulsions and Rucide appears in Table 2. A general tendency will be noted for persistency of emulsions to vary with concentration. However, the variation is not entirely direct, as trebling the concentration (e.g., raising from 4 to 12 per cent.) does not even double the protective period.

TABLE 1.—ANALYSES OF HAIR SAMPLES FROM SHOULDERS AND FLANKS OF ANIMALS SPRAYED WITH 4 PER CENT. DDT PREPARATIONS.*

Preparation Used	Days after Spraying †	Situation Sampled	Gamma DDT per gram Hair.
Kerosene emulsion ..	7	Shoulder ..	5,050
		Flank	964
Kerosene solution ..	18	Shoulder ..	1,118
		Flank	123
Kerosene solution ..	80	Shoulder ..	109
		Flank	74

* Analyses performed by Mr. R. F. Powning of the Division of Economic Entomology, by the dehydrochlorination method.

† The figures given for the cattle sampled at 80 days are at about the general level obtained when testing hair from untreated animals.

In the case of Rucide, persistency does not appear to increase with increasing concentration of spray between ½ per cent. and 3 per cent. p,p'-DDT.

TABLE 2.—RESULTS OF EXPERIMENTS WITH THE USE OF DDT EMULSIONS AND RUCIDE FOR THE PROTECTION OF CATTLE AGAINST THE BUFFALO FLY.

Concentration Percentage w/v p,p'-DDT.	Type of Emulsion.	Number of Cattle Sprayed	Protective Period (Days).*
12 ..	E. polybractea + E. dives var. C	38	21–28
	E. dives type	81	21+
4 ..	Solvent naphtha	141	14–21
		49	15+
		63	14–19
	Power kerosene	43	11+
		48	7–14
	E. dives type	30	14–21
3	Solvent naphtha	58	12
2 . ..	Solvent naphtha	30	9–14
0·2 . ..	E. dives type	31	Less than 7

Rucide.

Concentration Percentage w/v p,p'-DDT	Number of Cattle Sprayed	Protective Period (Days)
3 .. .	81	Less than 12
	62	Less than 16
2	37	9–15
0·5 .. .	70	10–17
	40	10–17

* "Protective period" is arbitrarily chosen as the number of days elapsing after spraying before the fly population is restored to more than a dozen per beast.

(b) Use of DDT Solutions as Light-mist Sprays.

(1) Preparations and equipment employed.—Solutions of DDT in power kerosene have been found quite suitable for use in this method. Ordinary household atomizers are the most suitable appliances for this method of spraying, as the preparation used is potentially harmful to the skin, and stronger atomizers would deliver a spray which would actually penetrate to the skin surface.

(2) Results of experiments.—Solutions of different concentrations were issued to various dairymen, with instructions to use as a light-mist spray on any cows coming into the bails carrying a considerable number of flies. Inspections were made at intervals to determine the degree of control achieved.

All flies present at the time of spraying that are caught in the mist are killed, and as the spray is directed on to the places where the flies cluster in the greatest numbers, a light deposit is left where it has a residual effect on flies alighting on the cows after spraying.

The exact degree of control achieved by this method of using DDT depends on the thoroughness of the spraying programme, and the individual dairyman's conception of what constitutes an infestation of economic significance. The initial reduction of a gross infestation necessitates the spraying of most of the herd, but after that has been done it is generally sufficient to spray only a few cows every day or so to keep down the numbers of flies. Generally farmers have little trouble in keeping infestations down to a level of less than a dozen flies per beast. Observations over two seasons have indicated that a quart of 4 per cent. DDT solution in power kerosene suffices to protect a herd of 30 cows for about a month. The exact concentration of spray used is not of great importance, except that with the lower concentrations greater quantities of spray are used, with increased risk of injury to the skin of the cattle.

(ii) *Use of DDT Primarily for Cattle Tick Control.*

Observations on this aspect of the problem are confined to experiments carried out at Rannes, Central Queensland, in February 1945. This work was done in collaboration with officers of the C.S.I.R. investigating the control of the cattle tick.

(a) *Preparations employed.*

Taubmans' Rucide, described in an earlier section, and an emulsion of DDT in solvent naphtha were tested.

(b) *Equipment employed.*

For the control of ticks on cattle it is imperative that the whole of the body surface should receive a thorough drenching with the toxicant. A plunge dip which will completely immerse the body of the largest beast, or else a spray race with numerous jets to cover all aspects of a beast passing through it, must be employed. Experiments were carried out with a plunge dip on Mr. J. L. Wilson's property at Rannes, and also with a Buzacott spray race on the adjoining property of Mr. C. Guest.

(c) *Results of experiments.*

(1) *Dipping experiment.*—The timber-lined vat was charged with 3,000 gallons of creek water, which was softened by the addition of 1/1000 by weight of " Calgon " $(NaPO_3)x$. One per cent. w/v Rucide was added on February 8, 1946, making the p,p′–DDT content ½ per cent. w/v.

On the day the dip was charged, 1,081 head, mostly 1–2 year old Herefords, were dipped. Tagged animals were introduced into the series of animals at regular intervals for detailed study of the effect of dipping on the tick population at different stages of the run. Before dipping, the fly population on these young stock was estimated to average about 100 flies per beast, with up to 300 on some animals. Differences in the behaviour of the flies from what is usual during dipping in arsenical preparations were observed. The flies did not

work back on to the undipped beasts, there was no tendency for them to gather round the entrance to the vat (where they often rest in numbers on the rails), and very few of them followed the cattle through to the draining pen. Most flies seen in the draining pen were strongly affected with DDT, and it appeared that the Rucide exerted a slight repellent effect while the hide was still wet, as flies alighting on the cattle made only momentary contact. Probably most of the flies were knocked down or rendered incapable of normal behaviour by a mist of fine droplets caused by the plunging of the beasts. Cattle inspected several hours after dipping were standing quietly in the shade, and no flies were seen.

On February 9, 650 animals were dipped, including a considerable number of full-grown Hereford steers and bulls. The average infestation on this mob was judged to be about 300 flies per beast, with up to 2,000–3,000 in some cases. Greater numbers of flies went through to the draining pen, but this may have been merely a result of the greater population of flies. The quietness of the cattle after dipping was in marked contrast with their restless behaviour before dipping, when the flies were apparently worrying them.

The animals tagged for tick examinations were kept in a separate paddock, and were available every few days for close study in yards. No flies were seen on any of them on or before the 7th day after dipping. A total of 3 flies was seen on the tagged beasts on the 10th day after dipping, and a few more on the 14th day, though still much less than 1 fly per beast. Frequent horseback inspection throughout this period showed that in all groups of dipped animals flies were absent, irrespective of age group or sex of the cattle, although on untreated cows on the same property there was an infestation estimated to average about 300 flies per beast. Five weeks after the original dipping the cattle were brought in again for dipping, and were reported to be still almost free of flies except for small numbers on a mob which had been grazing in a paddock alongside one containing heavily infested, untreated cows.

Reinfestation tests.—A mob of 1–2 year old Herefords which were dipped on February 8th were mixed with more than half their number of heavily infested, mature cows 5 days after treatment. The cows and the heifers were forced to mix with one another in this paddock, as there was only one watering place. When drafted out the following day the cows carried infestations very much smaller than when they had been introduced to the paddock. There were no flies to be seen on the treated heifers.

A similar deliberate reinfestation was performed on the 9th day after dipping. Drafting on the 10th day still revealed no flies on the heifers. There were fewer on the cows than when introduced into the paddock, but the reduction was not so high as on the first occasion.

A third reinfestation was carried out on the 14th day after dipping. cows were drafted out on the 15th day, when a light infestation seen on the treated heifers. When some of these were inspected for 5 hours after drafting, however, very few flies could be seen, so is likely that DDT was still active on at least some of the beasts.

Bioassay tests.—Clippings taken from the shoulders of the tagged beasts on the 5th and 7th days, and from both the shoulders and the flanks on the 10th and 14th days after dipping, were tested for their toxicity to the buffalo fly by the method described earlier.

Strong traces of DDT were found on the hair from the shoulders of most of the beasts on the 5th and 7th days. On the 10th day after dipping there were still fairly strong traces on the shoulders, but much less DDT on the samples from the flanks of all the beasts. The samples taken on the 14th day after dipping showed some DDT on the shoulders of 4 of the 10 beasts sampled, but only weak traces of DDT on the flanks of 2 of the same 4 beasts, with none on the remainder.

The life-cycle of the fly was estimated not to exceed 10 days under the hot conditions prevailing at the time of the tests, so that it appears highly probable that the protective period of the dipping lasted long enough to kill all flies emerging from the dung on the property on which the treated cattle grazed. Thus the initial fly population was entirely eliminated, and reinfestation was left to infiltration of flies from outside the boundaries. As the area oh which the treated cattle grazed was a compact block of approximately 15,000 acres, infiltration would be expected to be slow. This is an adequate explanation of the long period over which the cattle were observed to remain free of flies. The same reason has been advanced by Matthysse (1946) to account for the prolonged scarcity of horn flies on beef cattle sprayed in Florida with DDT preparations, and Peairs (1946) for the disappearance, after one treatment with DDT, of horn flies from a small herd of heifers in Virginia, which were effectively isolated from reinfestation.

(2) *Spray race experiment.*—The spray race was a Buzacott product, 36 feet in length, with rows of nozzles above and below the cattle on either side, the upper nozzles directed downwards and inwards, and the lower upwards and inwards. It was designed for continuous operation, the cattle walking from the forcing yard to the draining pen through the drenching spray.

(*a*) The first spray tested was a solvent naphtha emulsion containing 1 per cent. crude DDT (75–80 per cent. p,p'–DDT). On February 10 this was applied to 100 Australian Illawarra Shorthorn milkers, which, before spraying, were infested with about 50 flies per head. No flies were seen in the draining pen, and there was no accumulation of drowned flies in the gauze screens of the filters over the reservoir, as usually occurs when arsenical fluids are employed. During spraying operations about a dozen flies strongly affected by DDT were caught in an entomological net which was standing like a windsock downwind from the entrance to the race. Apparently the flies were caught in a mist of fine droplets at the mouth of the race, and immediately becoming affected with DDT, lost the power of directive flight, and were carried away downwind.

Cows were tagged at regular intervals during the spraying for detailed tick studies, and the tagged cows were run with the rest of the herd. The cows remained entirely free of flies for the first 9

days, after which small numbers, less than 1 per beast, could be seen, but it was reported that the infestation did not build up markedly during the 5 weeks following spraying. The dairy was 2,000 acres in area, adjoining the property on which the cattle were treated in the plunge dip, and so the failure of the flies to reappear on the cattle may be attributed to the same causes already suggested.

Bioassay tests.—Hair samples were taken from the shoulders of the milkers on the 3rd, 5th, and 8th days after spraying. There was fairly strong evidence of DDT on the shoulders of the cows on the 3rd and 5th days, but on the 8th day only one beast gave a positive test. The cows were particularly short in the coat, however, and in some cases only very small samples of hair were secured. This invalidates any comparison with the long-haired Herefords that were used in the dipping test.

(*b*) Also on February 10, 100 full-grown A.I.S. steers were sprayed with 1 per cent. Rucide ($\frac{1}{2}$ per cent. p,p'–DDT) prepared with rain water. The animals which were tagged at regular intervals during the spraying for detailed tick studies were transferred to the property where the dip was situated, and kept with the tagged Herefords from the dipping experiment. The remainder of the steers were retained on the same property as the milkers, but kept in a separate paddock.

Before spraying the steers carried an average infestation of about 100 flies per beast. The history of the fly population on both the tagged and untagged steers was the same as on the dipped animals.

Bioassay tests.—Hair samples were taken from the shoulders of the tagged steers on the 3rd and 5th days after spraying, and from both the shoulders and the flanks on the 8th and 12th days. The results of testing these with flies were comparable with those obtained for the dipped Herefords. In particular the more rapid disappearance of DDT from the flanks than from the shoulders was confirmed, as no flank samples were demonstrated to carry DDT, whereas shoulder samples from several of the beasts on the 8th and 12th days showed distinct traces of DDT. The hair of the steers was longer, on the average, than that of the cows sprayed with solvent naphtha emulsion, but not nearly as long as that of the Herefords.

4. Persistency of DDT on Cattle

When preparations of DDT are applied to inanimate surfaces, persistency in sufficient quantities to act as a contact poison to insects usually runs into months. For instance the haired surface of a piece of fresh cattle hide was sprayed with 1 per cent. DDT in alcohol in October 1943. During the week following spraying, the hide was repeatedly exposed to sunshine, and washed under a tap with an amount of water equivalent to many inches of rain, but for the rest of the period during which it was studied it was stored indoors. More than 8 months after treatment the hair was found still to be toxic to buffalo flies confined on it under a petri dish. There is an extensive literature concerning the prolonged persistency of DDT deposits on inanimate surfaces.

By contrast, the persistency of DDT on the living bovine is short, even considering the almost continuous exposure of the deposit to the effect of the elements. The persistency of DDT on cattle is known to depend to some extent on the type of preparation employed. For instance Matthysse (1946) states that deposits from suspensions are more persistent than deposits from emulsions.

The rate of application of sprays affects the persistency. Light mist-sprays of the type described on page 32 have very low persistency when compared with sprays which thoroughly drench the hide with a preparation of the same concentration of DDT. This simply amounts to a variation in the initial quantity of DDT applied to the hide (see below). Table 2 provides some evidence that persistency of emulsions is governed by the concentration of spray applied. Evidence that the same holds for solutions is provided by the following experiment:

Groups of 3 cows within the same herd were evenly sprayed with solutions of 1, 4, 8, 12, 16, and 20 per cent. w/v DDT in solvent naphtha at Malanda on May 15, 1945. As the cows from each treatment were running together, results could not be judged by fly population measurements. Instead, bioassay tests were made on hair samples taken from the shoulders of the beasts at intervals after spraying. Table 3 indicates quantitatively the relative progress of the DDT poisoning in the flies after one hour of exposure to the samples. It will be noted from the table that persistency appears to

TABLE 3.—TOXICITY TO BUFFALO FLIES OF HAIR SAMPLES FROM COWS SPRAYED WITH SOLVENT NAPHTHA SOLUTIONS OF DDT.*

Days after Spraying	W/V Percentage DDT in Solvent Naphtha					
	1	4	8	12	16	20
1	4	5	4	4	5	5
4	4	5	4	4	4	5
7	4	5	5	5	5	4
10	3	4	4	5	4	4
13	3	4	4	4	5	5
16	2	4	4	5	5	5
19	1	3	4	4	4	5
22	··	3	2	4	4	4
25	··	1	3	4	5	4
30	··	2	2	3	3	2
35	··	··	3	3	3	1
	··	··	—	3		
40	··	··	—	—	—	1
	··	··		2	3	1
	··	··		··		
45	··	··	··	—	1	
	··	··	··	2		
50	··	··	··	—		
	··	··	··		1	
55	··	··	··	—		—

*Ratings in the table illustrated the relative progress of the symptoms of DDT poisoning in the flies after one hour of continuous exposure to the samples Up to the 35th day the samples were bulked for testing, but for the remaining four sampling days the hair from the three cows in each treatment was tested separately.

diminish after the concentration of the spray increases beyond 12 per cent. The most likely explanation of this is that the sprays caused scalding which was observed to vary with the concentration of the DDT in the spray, and that desquamation, which was only very slight with the sprays of low concentration, was particularly severe on the cattle sprayed with 16 and 20 per cent. solution. It is possible that the scabs of epithelium working out through the hair carried away a proportion of the DDT in the case of the 16 and 20 per cent. sprays.

The samples taken the day following treatment showed that the hair from the 1 per cent. treatment affected the flies somewhat more slowly than the hair from the remaining treatments. This seems to suggest that somewhere between the 1 and 4 per cent. concentration complete coverage of the hair with DDT occurs.

5. Factors Responsible for Loss of DDT from Cattle

Early in the research consideration was given to the possible factors responsible for the comparatively rapid disappearance of DDT from cattle. Suggested factors were as follows: (a) Rainfall, (b) solar radiation, (c) detoxification by dust or mud, (d) rubbing and licking by the cattle, (e) natural shedding of hair, (f) absorption into the skin and hair, (g) removal or destruction by skin secretions, (h) removal by flakes of epithelium.

Factors (a) and (b).

Peairs (1946) attributes importance to rainfall. Matthysse (1946) also considers that rainfall and sunlight are of major importance in the removal of DDT from sprayed cattle, advising spraying under the belly as well as over the back, to ensure lasting protection from horn flies. Evidence has been advanced in previous sections to show that DDT disappears more rapidly from the flanks than from the shoulders. This is inconsistent with the theory that rain and solar radiation are major factors in the removal of DDT from cattle.

To obtain some check on the effect of these factors on the rate of disappearance of DDT from cattle, two pieces of hide from a freshly killed beast were sprayed with 4 per cent. DDT in power kerosene, and two with 4 per cent. DDT in power kerosene emulsion. One half of a hide from each pair was protected from rain by covering it with a shield of celluloid, which did not, however, impede the circulation of air. The other half was left unprotected. These hides were exposed to the weather on a roof at Malanda on April 28, 1944, the other member of each pair being stored indoors as control. One month after exposure both the portion exposed to sun and rain and that exposed to the sun only, of both solution- and emulsion-treated hides were as strong in their effect on buffalo flies (confined on them under petri dishes) as the corresponding controls. Only 15 points of rain had been recorded since the date of exposure, and the days had mostly been fine and warm.

Two months of exposure produced a noticeable weakening of the DDT on the completely exposed end of both hides compared with the appropriate control. The portions protected from the rain on each hide showed a higher concentration of DDT than those exposed to both

sun and rain, but the effects on the flies were noticeably weaker than those produced by the controls. The rainfall recorded since the previous test totalled 519 points, and there had been very little sunshine.

At $3\frac{1}{2}$ months these effects were somewhat accentuated, but there was still sufficient DDT on all the exposed hides to kill buffalo flies. A further 551 points of rain had been recorded since the previous tests, with a fair number of fine days.

Had similar preparations been applied to living cattle at the same time as the hides were treated, it is unlikely that even a trace of DDT would have been detected at the first examination one month after treatment, irrespective of the weather conditions to which the cattle had been subjected. Thus it appears that the effect of weather is quite insufficient to account for the rapid disappearance of DDT from living cattle.

Factor (c).

Detoxification of the DDT by dust or mud, either mechanical or chemical, may occur under certain circumstances, but DDT disappears from cattle grazing on unbroken swards of green paspalum, where both dust and mud are reduced to a minimum.

Factor (d).

Rubbing and licking by the cattle probably remove considerable quantities of DDT. The disappearance of the DDT faster from the flanks than from the shoulders may be accounted for by the animals rubbing against bushes and long grass. Whenever a beast lay down to rest, it would probably brush off some of the deposit.

Some beasts at least can reach a considerable area of the body surface with the tongue, from the butt of the tail to the base of the neck, in the course of the natural cleaning of the skin. It is not unlikely that licking, which is an exceedingly vigorous action, removes a considerable amount of DDT. In one experiment in which a DDT spray was applied to cattle in sufficient quantity to leave a white coating on the hair, the visible deposit was completely removed where the tongue had traversed the skin.

Factor (e).

Natural shedding of hair in small quantities is a continuous process (Duerden and Whitnall, 1930). At most times of the year it is insufficient to account for the rapid disappearance of DDT from cattle, though it may be a factor during the post-winter moult, which does not, however, extend over a very long period.

Factor (f).

Absorption into hair, if it occurred, would be detectable by chemical analysis of hair sampled after other methods had indicated there was no DDT left on the hide, but analyses do not point to this factor as a reason for the disappearance of DDT from cattle. Absorption into the skin is more likely to occur, but more difficult to detect.

Factor (g).

Removal or destruction by skin secretions is a factor still awaiting investigation. Kelly (1943) states that European breeds of cattle have almost lost the power of sweating. This would appear to rule out at least a mechanical effect from sweat. Little is known of the sebaceous material of cattle, but the continuous secretion of this may play a part in the removal of DDT.

Factor (h).

The mechanical effect of desquamation may be of some importance. The natural shedding of scurf would certainly remove the DDT from the immediate skin surface. However, it is doubtful whether scurf in working its way out through the hide, would scrape DDT off the hairs, though where scalding had been produced by a spray, large scabs working their way out through the hair may scrape off some DDT.

To sum up, most importance in the removal of DDT from cattle is attributed to the beasts' licking themselves, and rubbing against plants, the ground surface, etc. The effect of meteorological factors is secondary in importance. In addition there are factors associated with the skin physiology of the cattle which still await elucidation.

6. Discussion

As stated earlier in this paper, the problem of the control of the buffalo fly on cattle is closely bound up with the problem of the control of the cattle tick. Should DDT come into general use for the control of the tick, the control of the buffalo fly would be achieved automatically. In comparing the cost of using Rucide for the control of tick with that of arsenical dipping, this fact should be taken into account.

Where the use of arsenicals is continued, mustering for dipping affords a convenient opportunity for the application of the comparatively small dose of DDT required for the control of the fly. Cattle entering a dip with a wet deposit of DDT emulsion on their hides have been found by chemical analysis of hair samples to lose about 95 per cent. of the DDT in the dip.* Therefore the only practical place for spraying is in the draining pen, unless another crush is available, to which the cattle can be passed without trouble after dipping. No interference with the effectiveness of DDT sprays seems to result from spraying cattle that have only partially drained after dipping. Unfortunately in some cases draining pens are large enough to permit fairly free movement of the cattle, making it difficult for an operator to reach beasts in the centre. Better access can be had by erecting a " catwalk " of raised planks around the pen, which will enable a man to operate spraying gear over the fence, without interference to his movements by the posts or rails. Knapsack sprays with trigger-grip control of jet, and enough hose to permit one man to operate the pump while another manipulates the spraying nozzle have been found satisfactory in the comparatively small-scale experiments

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* Analyses performed by Mr. R. H. Hackman of the Division of Economic Entomology.

described herein, and many graziers have successfully used these and other hand-sprays for the treatment of beef cattle after dipping. However, for really large-scale work, motor-driven pumps are essential, and attention is now being given to the selection of suitable machinery for this purpose. A simplified spray race, with draining floor, and a few permanently-fixed jets to treat the upper parts of the animals is also a practical possibility.

As mustering for tick control is availed of for the application of sprays to control the buffalo fly, the spray applied should produce satisfactory control of the fly for the period of 3 weeks which usually elapses between dippings. Matthysse (1946) states that emulsions must contain at least 2·5 per cent. DDT to afford lasting protection against the horn fly. In the research on the buffalo fly, most attention has been given to emulsions containing 4 per cent. DDT, as it was found early in the investigations that this concentration very nearly fulfilled the requirement of affording protection for the period of 3 weeks between dippings for the control of the tick. Increasing the protective period by even a week involves an inordinate increase in the concentration of the spray used. Possibly slightly lower concentrations of emulsions would prove satisfactory, but even treatment with 4 per cent. preparations sometimes permits the re-establishment of a considerable population during the third week after spraying. Rucide gives satisfactory results at 1 per cent. concentration (½ per cent. p,p′–DDT), ·resulting in the cattle remaining virtually free of flies for about a fortnight. Increasing the concentration of the Rucide does not appear to enhance protective periods as with emulsions. Matthysse (1946) found that no gain resulted from increasing the concentration of suspensions above 1 per cent.

For large-scale use of DDT against the buffalo fly, Rucide is unquestionably the most suitable preparation yet available. For the small owner, however, emulsions have the advantage that any fraction may be drawn from a supply of concentrate without detriment to the quality of the remainder. On the other hand the stratification of solidified Rucide requires that the whole of a batch should be melted before a portion is removed. The remelting of the bulk supply creates a risk of wastage of material.

The partial spraying (i.e., spraying over shoulders only) of cattle with 1 per cent. Rucide is the cheapest method of buffalo fly control. About 3 gallons of spray are required for one treatment of 100 head. During the course of the average buffalo fly season (usually about 5 months), approximately 7 sprayings are required. The present price of Rucide (1 lb. lots) is 8s. 6d. per lb., so that the cost of treating 100 head against buffalo fly in a typical district is about 19s. per annum.

Dairy farmers may use either traps or sprays to keep their milking cows free from buffalo flies, but partial spraying of cattle for buffalo fly control is so cheap and effective that the use of traps is no longer considered economical. The initial cost of a trap is £25–£30, which, quite apart from depreciation and maintenance, is equivalent to the cost of sufficient spray materials for the protection of 100 head (more than the usual number of cows in a milking herd) for between 20 and 30 years. Furthermore there are usually dry cows and other stock on a dairy which cannot be made to use a trap, and for the protection

of which spraying gear and DDT must be purchased irrespective of what is done for the protection of the milking herd. Partial spraying may therefore be advised as the most generally useful method of controlling the buffalo fly.

As described in Section 3 (ii) of this paper, there is good reason to believe that the larger the area of land over which simultaneous treatment of all cattle with DDT is carried out, the more effective and prolonged is the control of the buffalo fly. Thus it appears logical to conclude that the most economical and effective method of buffalo fly control in closely subdivided districts would be the simultaneous spraying with DDT preparations of all cattle over wide areas.

7. Acknowledgments

Appreciation is expressed of the cooperation afforded by farmers, too numerous to mention individually, who have assisted by making their herds available for purposes of experiments.

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The Use of DDT in Dips to Control Cattle Tick

By L. F. Hitchcock, M.Sc.,* and I. M. Mackerras, B.Sc., M.B., Ch.M.*

Summary.

Field experiments are reported in which good control of cattle tick (*Boophilus microplus* Canest.) was obtained with dips charged with ½ per cent. DDT in a water-miscible preparation.

Residual activity lasted between one and two weeks, during which time the cattle appeared to act as poisoned baits clearing the pastures of larval ticks.

1. Introduction

The dipping observations here reported were planned by the writers, but were carried out in various parts of Queensland by several members of the Division of Economic Entomology, namely, at Rannes, by L. F. Hitchcock, W. J. Roulston, and W. R. Horne; at Gracemere, by W. J. Roulston; at Toomba, by W. J. Roulston and R. W. Kerr; and at Innisfail, by G. J. Snowball, R. W. Kerr, A. Mills, and R. Meyers.

During the past four or five years, owners in parts of Queensland had been experiencing increasing difficulty in controlling ticks, even with fortnightly dipping in ordinary arsenical solutions, while many cattle moving south were being held up for repeated dippings at cleansing stations. It seemed evident that resistance to arsenic was spreading in the tick population, and the search for an alternative killing agent therefore became urgent. Evidence from abroad (unpublished O.S.R.D. reports) and preliminary experiments in Queensland had shown that emulsions containing DDT were efficient in killing the cattle tick (*Boophilus microplus*) in all its parasitic stages, but a stumbling block to further progress was the difficulty in obtaining a preparation which would remain stable in a dip and could be used safely on a large scale. At this stage Messrs. Taubmans Ltd., Sydney, developed a water-miscible preparation, which they registered under the name " Rucide," and which gave some promise of fulfilling requirements. Owing to the energetic co-operation of Mr. J. L. Wilson, Chairman of the Cattle Committee of the United Graziers Association of Queensland, and his willingness to risk losses of cattle to test the efficacy of DDT in dips, we were able to begin field trials much more quickly than would otherwise have been possible.

The work is not complete, but the results of the field experiments have been so good that a preliminary report is felt to be justified.

There are three separate phases in the control of cattle tick in Queensland:—

> (i) Control of ticks on grazing and dairying properties. The aim is not to exterminate the ticks, but to reduce their population to a level at which the minimum of damage is caused, while permitting enough transmission of piroplasmosis for cattle to maintain their immunity.

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(ii) Cleansing cattle for movement from infested to clean country, so that spread of infestation in Australia will be retarded.

(iii) Reclaiming infested country by a campaign of eradication.

We are concerned here only with the first of the three. Cleansing experiments are being undertaken, and the residual activity of DDT gives it obvious advantages for eradication of ticks, but neither will be considered further in the present paper.

Our thanks are especially due to Messrs. J. L. Wilson and C. McDouall (Rannes), Alister Archer (Gracemere), E. E. D. White and H. McKinnon (Toomba), and W. D. Davies and A. Stoter (Innisfail) for their co-operation and help in the field work, and to Messrs. Taubmans Ltd., Sydney, for the gift of part of the Rucide used.

2. Preliminary Experiments

Several experiments were carried out on small groups of cattle in 1944 and 1945 by one of us (L.F.H.). The cattle were mostly sprayed individually, usually with a power sprayer, and DDT emulsions were used in most experiments, though solutions were occasionally employed. Their purpose was to determine the toxicity of DDT to ticks, but they also provided guidance for the later work with Rucide, so the results may be reviewed briefly here:—

(i) Using 0·5 per cent. DDT in alcohol on artificially infested cattle, semi-engorged nymphs were found to be relatively resistant, and all other parasitic stages highly susceptible. This observation has been confirmed in later field work, the nymphs being the slowest to die, and any survivors maturing having been in the nymphal stages when dipped.

(ii) On naturally infested cattle sprayed with emulsions, 0·5 per cent. DDT gave a good kill of ticks, 1 per cent. a very good kill, and 2 per cent. practically consistently a complete kill in 68 animals tested. Protective periods, during which larval ticks failed to attach, were:—

0·5 per cent. DDT—7 days.
1 per cent. DDT—9–10 days.
2 per cent. DDT—10–12 days.

(iii) In examining the effect of season, ten animals were sprayed with 2 per cent. DDT emulsion in September, December, April, and July. The kill was practically 100 per cent., and the protective period 11–12 days at all seasons.

(iv) In a preliminary run with Rucide, spraying only one animal at each strength, the following results were obtained:—

Strength.	Kill.	Protective Period.
2 per cent. DDT ..	complete ..	15 days.
1 per cent. DDT ..	complete ..	12 days.
0·5 per cent. DDT ..	very good ..	10 days.
0·25 per cent. DDT ..	good ..	7 days.

In view of these results, and because dipping was expected to wet the cattle more efficiently than spraying, 0·5 per cent. DDT was chosen for the first dipping experiment.

Although there was no reason from the literature (unpublished O.S.R.D. reports; Orr and Mott, 1945) to expect that DDT would be toxic to cattle at the concentrations proposed, it was considered wise to carry out some experiments under Australian conditions. A well-grown beast might retain a gallon of fluid on the skin and hair, so it might conceivably absorb about 20 g. of DDT, or roughly 50 mg. per kilogram, from a single dipping in 0·5 per cent. DDT. Dipping might be monthly. Three experiments were set up, in which dosage considerably exceeded these amounts:—

    (i) Two animals were given an aqueous suspension of DDT by stomach tube fortnightly; one received seven 25-g. doses in 14 weeks, the other three 50-g. doses in 6 weeks; both remained well.

    (ii) One animal was given 50 g. DDT in 200 ml. eucalyptus oil and liquid paraffin *per os* fortnightly for six months, and a control received the oil mixture without DDT; both remained well.

    (iii) Eighteen young Hereford steers of mean weight 548 lb. have been inuncted along the back weekly with 22·5 g. DDT in 300 ml. peanut oil. These animals were intended to be maintained on a high plane of nutrition in Queensland, while a parallel series under the care of Dr. A. W. Turner in Melbourne were to be kept on a low plane; owing to season, the nutrition of the Queensland group is only fair. The experiment is to run three years, with a group from each series killed for post-mortem examination at the end of each year. To the end of 33 weeks, no evidence of DDT poisoning has been seen.

3. Preparation of Rucide Dips

Rucide consists of 50 per cent. p,p′–DDT incorporated in a "solubilizer." It is a solid, with something of the appearance and consistency of lard, and must be melted by heat before mixing with water. The procedure is to melt the Rucide, with constant stirring, over a slow fire, pour it slowly into a drum of soft or softened water (again with vigorous stirring) to make a concentrate of from 4 to 12 per cent., and then tip the concentrate into the water in the dip. The resulting suspension is exceedingly fine and stable, almost of colloidal quality, though it does tend to settle out slowly with time.

Certain points in the use of Rucide require brief mention:—

    (i) Heating must be slow and careful. Rucide melts at about 100°C., and the temperature does not rise much above that level until a certain amount of water present has been driven off in fine bubbles. Normally it forms a clear brown solution. When all the water is driven off decomposition occurs at about 150°C. The solution darkens, and will no longer mix with water (Powning,

unpublished). Local over-heating will produce the same effect in part of the material, so the need for caution is apparent.

(ii) Rucide will not mix with hard waters, and a softener must be added if ordinary soap will not lather readily in the water. We have had excellent results with " Calgon " (sodium hexametaphosphate) at the rate of 1 oz. Calgon per degree of hardness per 100 gallons of the water. Even with soft waters we have the impression that small amounts of Calgon (e.g., 1 in 10,000) improves the fineness and stability of the suspension.

(iii) The strength of the initial concentrate may be varied as desired, but should not exceed about 12 per cent. (Powning, unpublished). Thus, the contents of a 56-lb. pail may be poured into a 44-gallon drum of water, or it may be divided between two drums. Concentrates appear to remain stable for some hours, but the general practice is to have the drum at the entrance or exit of the dip and pour it in as soon as mixing is complete.

(iv) It is not necessary to be over-meticulous in cleaning out a dip preparatory to filling with Rucide. Excess debris and deposit should, of course, be removed, but there is evidence that a moderate layer of silt on the bottom is beneficial, in that it prevents caking of DDT that settles out, and ensures that it will re-disperse readily when thoroughly stirred by the first few cattle to be put through.

It is unfortunate that, up to the time of writing, we have not been able to use chemical analysis to determine the actual amount of DDT present in the dips. Consequently, the amount of Rucide to be added when topping up after use can only be calculated from volume on the assumption that the remaining fluid still contains $0 \cdot 5$ per cent. DDT, while the activity of the fluid can only be assessed by its actual effect on tick-infested animals. These limitations prevent us from offering collateral evidence on the results obtained; but, after all, it is the practical effect of dipping in which the grazier is interested, so we feel that the work does not lose so much by being limited to an examination of that effect.

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4. The Dipping Experiments

Observation of the general effect of dipping is easy, but quantitative estimation is difficult, chiefly because the tick population cannot be determined by any method short of placing control animals in a stall and collecting the adults, as they fall, over the ensuing five weeks. Consequently, the percentage kill of ticks cannot be recorded except in very general terms. Data were collected in three categories:—

(i) The general infestation of the herd was estimated prior to dipping as light, moderate, or heavy, on the basis of numbers of ticks observable and their distribution on the body. Subsequent to dipping, surviving ticks could be

counted individually if the result was satisfactory, but the kill was usually recorded as complete, nearly complete, or incomplete.

(ii) Close examination of a sample series from the herd was made to determine the stages of development of the ticks present prior to dipping and also at various periods after dipping. These stages were classed according to Table 1 (from Hitchcock and Horne, unpublished), and the stage at dipping was calculated back for survivors after dipping by means of the same table. Results were recorded, not as numbers of ticks found, but as numbers of animals in the group on which the different stages were found.

(iii) Samples of the herd were searched for re-infestation at various periods after dipping, the results being recorded as in (ii).

TABLE 1.—STAGES OF *Boophilus microplus.*

(*Age in days from attachment.*)

	Larvae				Nymphs				Adults		
	Young	Medium Sized	Semi engorged	Engorged	Young	Medium Sized	Semi engorged	Engorged	Young	Medium Sized	Engorged
Minimum				3½	4½	7½	9	11	13	17	19
Mean	1 2	3	4	5	7	10	11	14	17	20	23
Maximum				8½	13	15	17	20			35

(*a*) *Rannes.*

This property, owned by Mr. J. L. Wilson and managed by Mr. C. McDouall, is 55 miles south-west of Rockhampton. On February 8, 1946, a dip was filled with 3,000 gallons of 1 per cent Rucide (0·5 per cent. DDT), the water being softened with 1:1,000 Calgon. On February 8 and 9, 1,800 head of Hereford and Shorthorn cattle, comprising 400 grown bullocks and bulls, 500 breeders, and 900 mixed cattle 9 to 24 months old, were dipped, animals for subsequent examination being tagged at intervals throughout the dipping.

Tick infestation was light, and all stages were present at the time of dipping. Subsequent examinations showed that the kill was slow, reaching a maximum in 5–6 days, but was good, most animals being completely clean, and the remainder having very few survivors, the maximum being less than ten. The protective period for 45 animals checked ranged from 6 to 10½ days, and averaged 8 days. These cattle were dipped again by Mr. McDouall five weeks later, when carrying an extremely light tick infestation.

On February 23, 1946, the dip was stirred but not topped up, and 105 head were dipped. The initial infestation and results were similar to the above.

Between February 8 and May 3, approximately 5,000 head of cattle were dipped, and 4,000 gallons of 0·5 per cent. DDT suspension added in topping up, about 2,000 gallons of fluid remaining in the dip at that time. Ticks were very scarce throughout this period.

On May 5, as sufficiently infested stock were not available on Rannes, a neighbour, Mr. C. G. Guest, kindly allowed us to put 58 of his milkers and two bulls through the dip as a check. It had last been used on May 3, when it was topped up with 1,000 gallons DDT suspension, and 1,000 bullocks were dipped. Mr. Guest's cattle carried a light tick infestation, with all stages present. Subsequently, only six surviving ticks could be discovered on the 60 animals. The protective period before re-infestation with larvae was fifteen days.

To July 16, five months after the dip was charged, a total of 5,400 cattle had been dipped on Rannes, and 7,600 gallons of 1 per cent. Rucide had been added, including the original charging. Ticks were still extremely scarce, and no cattle had been dipped since May. On July 17, 70 bullocks belonging to Mr. Donovan, another neighbour, were put through the dip. The owner had had difficulty in controlling the ticks with arsenic, and Mr. McDouall stated that all stages were present at the time of dipping. They were examined on July 21 by Mr. Roulston, who reported that the cattle were clean and no surviving ticks could be discovered.

Recently, we have seen a mob of weaners, which were put through this dip on October 16. They showed numerous dead remains but no live ticks, indicating that the suspension was still active.

(b) *Toomba.*

Toomba is owned by Mr. E. E. D. White and managed by Mr. H. McKinnon; it is situated 70 miles north-west of Charters Towers in a well-watered basin. Ticks had been consistently heavy, and for the past four or five years increasing difficulty had been experienced in controlling them. Prior to the experiment, the station milkers had been dipped in arsenic weekly, but still carried heavy infestations.

On May 29, 1946, a dip was charged with 2,400 gallons of 1 per cent. Rucide; the water was soft, so no Calgon was added. On May 30, 176 heifers and 31 milkers and calves were dipped. The heifers had been dipped in arsenic three weeks previously, and carried a light-moderate infestation of all stages of ticks. The cows and calves had been dipped one week previously, and carried a heavy infestation of all stages; their hides were raw, thickened, and scabby.

Of twenty heifers kept back for close examination, nine showed surviving ticks seven days after dipping. These had been in the nymphal stage when dipped, and the maximum number found on a single beast was ten. Re-infesting larvae were first detected on the eighteenth day after dipping.

The cows showed a few surviving ticks on the head, and in one instance there was a line along the jaw, above which ticks were alive, while below it they were dead. These cows were very experienced in being dipped, and it was suggested that they managed to keep their heads more or less clear of the fluid. Apart from this, the kill on the cows and calves seemed to be complete. The protective period was twelve days.

From May 30 to September 14, 5,735 head were dipped, and 2,540 gallons of 1 per cent. Rucide added. Throughout this period ticks remained scarce. Cattle on the property were examined by Mr. Kerr on September 4–15, the results, with dipping information supplied by Mr. McKinnon, being set out in Table 2.

TABLE 2.—TOOMBA DIPPING RECORD.

Paddock.	Number.	Previous Dippings in DDT	Last Dipping to Examination (Weeks)	Infestation
Horse	25	May 30, July 17 ..	7	Very light
Long Pocket ..	186	Last dipping, Aug. 12	3½	Clean
Canal	796	Last dipping : most July 11–17, a few June 3	7- 8⎫ ⎬ 14⎭	Very light, a few moderate
Oakey	147	May 31, July 11 ..	9	Very light
Lake	204	May 31, July 11 ..	9	Light, a few moderate
Nine Mile ..	105	July 3 only ..	10½	Moderate
Controls " Myola "	200	Dipped in arsenic, some three weeks, some six weeks previously		Light to heavy, majority moderate

On September 14 the dip was topped up with 560 gallons 0·5 per cent. DDT, and the 200 Myola bullocks, shown as controls in Table 2 and kindly made available by the owner, Mr. W. Jackson, were put through. It was only possible to examine them once after dipping, on the eighteenth day, when they were found to be clean, although they had been returned to Myola, where ticks had remained active during the period under review (June to September inclusive) and DDT had not been used.

(c) Gracemere.

Gracemere, owned by Archer Bros., is within a few miles of Rockhampton. A dip of 1,600 gallons capacity was filled on July 23, 1946. The available water was extremely hard (59 grains per gallon, recorded as $CaCO_3$), and the dip was charged with 168 lb. of Rucide and 40 lb. of Calgon. The suspension formed appeared to be of satisfactory fineness and stability.

On July 24, 182 head of mixed cattle, carrying a light to light-moderate infestation of ticks in all stages, .were dipped. Kill was recorded as complete, except for an occasional engorged or semi-engorged nymph, and one or two adult ticks resulting from these nymphs were found on each of ten animals of 36 re-examined on the 22nd day. Re-infesting larvae were found on the 11th, 14th and 22nd days, but none beyond the medium-sized larval stage even at the last examination.

On July 24, two bullocks which had been used as controls for earlier spraying experiments and had not been dipped for several months were also dipped. They carried heavy infestations, and had a thick matted winter coat of hair carrying a lot of scurf and skin debris. The kill was incomplete, between 50 to 100 nymphs and adults surviving on each animal. One was re-dipped on July 29. On August 15, 22 days after the original dipping, the re-dipped animal was clean, and the other carried three adult ticks only.

Other cattle were dipped in the interim, and by September 5 the fluid in the dip was down to about 800 gallons. The suspension appeared normal, and was topped up with 450 gallons of water, 45 lb. Rucide, and 11½ lb. Calgon. One hundred and thirty-nine animals of the mob dipped six weeks previously were examined and dipped. They carried a light to moderate infestation of ticks in all stages. Six days after dipping 15 of the 21 kept for close examination were clean, seven showed one to four survivors, and one very large animal had a numbers of survivors on the shoulders. The protective period was not determined.

During this period, July to September, undipped controls had moderate to heavy infestations with all stages of ticks.

(d) Innisfail.

The dip used was at Goondi, within 5 miles of the town. It is owned by Mr. W. D. Davies, and the cattle dipped were his beef herd, and dairy cows belonging to Mr. A. Stoter. The dip was charged on August 4, 1946, with 1,860 gallons of 0·9 per cent. Rucide. The water was fairly soft, but 1:4,000 Calgon was added as a precaution.

On August 6, 156 head were dipped, comprising 26 dairy cows and two bulls, carrying a moderate infestation, and 128 heavily infested beef cattle of mixed ages and sexes; all stages of ticks were present.

Individual surviving ticks were found on about half the dairy cattle. The first re-infesting larvae were observed on the fifteenth day, and the first engorged adult resulting from re-infestation was found on the 35th day. On September 17, six weeks after dipping, they were considered by the owner and the Stock Inspector (Mr. J. Littleton) to be sufficiently infested to justify re-dipping. Owing to a misunderstanding, they were then sprayed with arsenic.

The beef cattle were in three mobs, one of which was available for regular examination. Practically all ticks were killed on this mob. only an occasional survivor being found at later examinations, while the protective period was estimated at fourteen days. When the cattle were put together for re-dipping on September 24, seven weeks after the first dipping, one mob (cows and calves in poor condition) was heavily infested, and the owner considered he should have dipped them a fortnight earlier; the second were lightly infested; and the third so lightly infested that dipping might with advantage have been postponed. All were re-dipped, with satisfactory control of ticks (Table 3) and a protective period of seven to nine days.

5. Discussion

These experiments were carried out over a wide range of country and tick populations from North to Central Queensland; and the Queensland Department of Agriculture and Stock has made observations with essentially similar results near Toogoolawah in South Queensland. Ticks on some of the properties were apparently " normal," but those on Toomba (and probably on Mr. Donovan's cattle) were " arsenic resistant."

Much remains to be done, particularly on the practical interval between dippings, the long-range effects of DDT on the populations of larval ticks in the pastures, and the balance between tick control and maintenance of immunity to piroplasmosis; but enough information has been obtained to give considerable hope for the future. The collective results of the experiments to date may therefore be reviewed briefly here as a starting point for future work.

(a) Effect on the Cattle.

The blandness of the DDT dipping in contrast to arsenic has been a striking feature to the owners of the cattle. Cattle rest and feed quietly after dipping, show no immediate irritation or later scalding, and a rapid improvement in the condition of their coats has been observed. It was also noted at Gracemere that they foraged more freely and widely after DDT dipping than after arsenic. No information is yet available about weights of beef cattle or butter-fat production of dairy cows.

(b) The Kill of Ticks.

As in the earlier spraying experiments, kill was slow, taking five to seven days to reach a maximum. Larvae and young nymphs disappeared within the first two days, and most adults by the fifth day. It is difficult to be sure whether the large adults were all killed, or whether some dropped off naturally. Engorged adults collected after spraying had usually failed to lay viable eggs, but a few kept at high humidity did (Horne, unpublished). The last to die were the engorging nymphs, and all the survivors subsequently found developing were in this stage when dipped.

It is difficult to estimate the percentage kill. However, no cattle were used for observation if their infestation was classified as less than " light " and unless all stages of the ticks could be readily found. Ticks are cryptic animals, except when approaching full growth, and surprisingly high populations are necessary even to qualify for the designation " light." Legg and Foran (1929) made a series of counts by stalling their animals and collected the adults as they matured. Their lowest count for an untreated control classed as " light " was 666, and the mean of ten " light " infestations was 3,489. We may safely conclude, therefore, that few if any animals in our series had less than 1,000 ticks on them prior to dipping.

One week after dipping in DDT, the animals in the groups kept for close examination fell into three classes: those on which kill was complete; those on which a very few (always less than ten, and usually only one or two) ticks survived and grew; and those on which

from about 20 to 100 ticks in various stages survived, and the kill was regarded as "incomplete." The distribution of these groups in these experiments is shown in Table 3.

TABLE 3.—THE KILL OF TICKS WITH DDT ONE WEEK AFTER DIPPING.

Experiment.	Original Infestation.	Number of Beasts Examined.	Tick Kill		
			Complete	Nearly Complete	Incomplete.
Rannes 1 ..	Light	45	30	15	..
Rannes 2 ..	Light	30	27	3	..
Rannes 3 ..	? Light	6	6	..	..
Toomba 1 ..	Light-moderate to heavy	25	14	10	1
Toomba 2 ..	Moderate	20	20	..	..
Gracemere 1 ..	Light-moderate to heavy	36	24	10	2
Gracemere 2 ..	Light to moderate ..	21	10	10	1
Innisfail 1 ..	Heavy	23	13	10	..
Innisfail 2 ..	Moderate	12	9	3	..
Total ..	Light to heavy ..	218	153	61	4

The kill with 0·5 per cent. DDT was therefore complete on 70 per cent. of animals examined carefully, less than 100 but more than 99 per cent. on 28 per cent. of the animals, and more than 90 per cent. in the remaining 2 per cent. when allowance is made for the heavy infestations they carried.*

(c) *Protective Period.*

The most remarkable feature of DDT dipping is that, unlike arsenic, the residue kills all invading larval ticks for several days after dipping. Cattle were seached for live larvae very carefully, and the day on which they first appeared was recorded. They are extremely difficult to see, so the results were checked by later re-examination at times when the re-infesting ticks would have grown sufficiently to be found with reasonable certainty. The day of attachment was then calculated from Table 1. The results agreed satisfactorily with the earlier observations, so it may be taken that the protective periods recorded were reasonably accurate.

The mean protective periods recorded varied from eight and a half to more than eighteen days.†

* Two of the "failures" were suspected to be due to incomplete immersion. One of the four was re-dipped and cleansed of ticks five days after the first dipping, and the other three became practically clean as ticks dropped off during the period of protection from re-infestation.
† Mulhearn (Queensland Department of Agriculture and Stock), using sprayed cattle and a different method of observation, has had shorter complete protection times, but his results still indicate an enormous destruction of re-infesting larvae.

These results require qualification in two respects. In the first place, the season was bad in some areas, and natural re-infestation may have been retarded. This applied to the first observation at Toomba, where frosts inhibited attachment of larval ticks on controls for three days during the critical second week of observations, and it may have applied, too, to Rannes. It did not hold for Gracemere, where controls became re-infested normally, nor to Innisfail, where the tick population was high and active, nor probably to the second observation at Toomba. On the other hand, there is evidence that many re-attaching larvae die, and that those first seen do not necessarily survive. The effective protective period may therefore be longer than we have recorded.

(d) Interval Between Dippings.

With a protective period of ten days and a developmental period of 22 days, a dipping interval of about four weeks might be considered necessary. However, the rate of re-infestation has, except for one group of Mr. Davies' cattle, always been slower than might have been expected, and the interval to the second dipping has been more of the order of six to seven weeks. Even then, most of the cattle have been too lightly infested for us to attempt to record the kill of ticks.

There may be three reasons for this prolongation of the interval to the second dipping:—

   (i) The dry (and, for part of the time, cold) season may have retarded re-infestation. This factor could have contributed to the result at Rannes, and doubtfully contributed at Gracemere and Toomba, but not at Innisfail.

  (ii) The effective protective time, during which most of the ticks attaching died, may have been longer than the complete protective time recorded. There is some evidence from post-dipping counts to favour this, and it seems likely (and reasonable) that the period of complete protection is succeeded by one of partial protection during which the DDT is gradually disappearing from the beast.

 (iii) While the cattle are still poisonous to ticks they act as poison baits, collecting the larval ticks from the pastures, and in this way they may produce a profound effect on the general tick population. We have as yet no accurate means to measure density of larval ticks on pastures, but we have a strong impression that the population is greatly reduced, and suspect that this may be the most important effect of all. The subsequent history of these cattle supports the suspicion.

The intervals between the second and subsequent dipping have generally been longer than those between the first and second. At Rannes we have never since the first dipping been able to find sufficient ticks to record kill, and have had to depend on the loan of neighbours' cattle to make observations; at the last inspection the Rannes cattle had not been dipped for three months. The position at Toomba is shown in Table 2, but the other experiments have not yet run sufficiently long to give information. Again, the dry season may have

played a part, though the presence of ticks on neighbouring properties makes it difficult to attribute the whole effect to season. In general, we are led to hope that, once control is well established (perhaps in bad areas by two dippings at fairly short intervals), the periods between dippings with DDT in seasons favourable to ticks may be two months or more.

(e) Stability of the DDT Suspension.

These dips have been used in accordance with normal practice; that is to say, they were charged, and then topped up from time to time with fluid to replace that removed by the dipped animals. This usually lay between ¾ and 1 gallon per head, and possibly some additional fluid was lost by leakage or evaporation. We do not yet know whether any significant changes of DDT content have occurred during the periods of usage, standing, and topping up with 1 per cent. Rucide. Judging by appearance, the suspension seems very gradually to settle out on standing, but to mix again readily (especially if there is a little silt in the bottom of the dip) when a few head are put through to stir it before dipping the main mob. On present indications, this would seem to be a sound precaution, even with freshly made suspensions.

Our longest experience so far has been for eight months at Rannes, at the end of which time the fluid appears to have been as active as it was at the beginning. At Innisfail, the fluid was active after six weeks' standing without topping up, but in all other observations fresh suspension was added before dipping, usually amounting to a fifth to one-third of the total volume of fluid. Thus, at the last detailed Rannes observations, the dip had not been used for three months, 500 gallons of fresh suspension were added to the 1,500 gallons of old suspension already present, and the cattle were then dipped.

So far as present experience goes, no term can be set to the duration of activity of this suspension.

(f) The Practicability of Dipping in DDT Suspensions.

DDT suspensions have certain advantages and disadvantages as dipping fluids. The advantages are:—

Efficiency in controlling ticks, whether arsenic-resistant or not.

Prolongation of interval between dippings, as compared with arsenic. (It has been estimated that dipping costs 2½d. per head for mustering and handling and ½d. per head for wear and tear of yards, in addition to the costs of materials and labour to fill the dip.)

Not only control but probably extermination of buffalo fly on the property (Norris, 1947; Snowball, unpublished); probably also control of lice, and possibly of sandflies (Simuliidae and Ceratopogonidae) and bushflies (*Musca vetustissima*).

Absence of irritation or scalding, so that cattle will rest contentedly, or can be worked freely, immediately after dipping.

> A probable, but as yet unassessed, improvement in condition and productivity resulting from a combination of these four effects.

The disadvantages are:—

> It costs about 4½d. per head for materials to dip in Rucide as compared with ½d. per head for arsenic.

> The suspension is more troublesome to prepare than an arsenic solution, and requires considerably more care and attention to detail.

The owner must weigh costs against benefits, and decide whether he is prepared to give the DDT suspension the intelligent handling it requires. In our present state of knowledge, we would unequivocally recommend the use of the suspension in areas where ticks are difficult to kill with arsenic, and buffalo flies are prevalent. In other areas, the balance is more delicate, and more experience is needed to decide whether the over-all benefits from DDT dipping outweigh the cheapness of arsenic dipping *plus* partial spraying with DDT (Norris, 1947) to control buffalo flies.

6. References

Legg, J., and Foran, J. L. (1929).—Some experiments on the treatment of tick-infested cattle with arsenical dipping fluids. *Proc. Roy. Soc. Qld.* 41: 83-120.

Norris, K. R. (1947). The use of DDT for the control of the buffalo fly (*Siphona exigua* (de Meijere)). This *Journal*, p. 25.

Orr, L. W., and Mott, L. O. (1945).—The effects of DDT administered orally to cows, horses, and sheep. *J. Econ. Ent.* 38: 428-32.

Addendum (February, 1947)

Several developments have occurred since the above was written:

(i) The long-range toxicity experiment mentioned on page 45 has now run for 51 weeks, without the cattle showing any detectable ill-effects.

(ii) In charging the dip, it is satisfactory to pour the drum of molten Rucide slowly directly into the vat, while the water is stirred vigorously with a paddle, thus saving the trouble of making a preliminary concentrate.

(iii) Analyses indicate that, after use, the DDT content of the dip often falls to 0·35 per cent. or less. In topping up, therefore, it is recommended that not more than 500 gallons of water be added for each 56 lb. drum of Rucide. If there is any doubt that the dip is killing ticks efficiently, samples taken *immediately* after stirring by about twenty head of cattle should be sent for analysis.

(iv) The dips mentioned in the paper have continued to give good control of ticks, while buffalo flies have remained difficult or impossible to find. Dips charged with 0·5 per cent. DDT have now been studied also at Ingham, Townsville, and Ayr, with essentially similar results to those recorded in the paper.

The Persistence of DDT on Cattle

By R. H. Hackman, M.Sc.*

Summary.

Evidence is brought forward to show that the most important factor causing the removal of DDT from cattle sprayed with DDT preparations is licking, either by the animal concerned, or by another animal. Other factors have been investigated, viz. solar radiation and rain, growth and loss of hair, rubbing, production of skin secretions, absorption into the skin and hair and flaking of the epithelium, but these have been shown to play a minor role. It is concluded that the amount of DDT ingested by licking would not be sufficient to produce toxic symptoms.

1. Introduction

DDT† has been shown to be a very valuable insecticide for the control of the major cattle pests, viz. cattle tick, buffalo fly, horn fly, etc. in Australia, the U.S.A., and South Africa. The frequency of application of DDT preparations for the effective control of these pests depends upon the persistence of the DDT on the animals, and the longer the period of persistence, the lower is the cost of control.

Field tests have shown that deposits of DDT on living animal surfaces do not have the lasting effect expected from tests on household insects. On non-living surfaces, deposits have been reported to be effective over a period of twelve months or more, but under field conditions, deposits on living cattle surfaces remain effective only for from one to two weeks for preparations containing up to 2 per cent. DDT. Many factors, viz. production of skin secretions, growth and loss of hair, leaching action of rain, disturbances by body movement and wind, friction against external objects, and licking, which combine to keep the coat clean, may play a part in the removal of DDT. To these as possible causes for the removal of DDT must be added breakdown under solar radiation, absorption into the skin and hair, and removal by flaking of the epithelium.

Three methods are available for measuring persistence:

(a) chemical analysis of extracts from clipped hair;

(b) bioassay of clipped hair samples; and

(c) following the trend of the pest population in the field.

Chemical analyses are useful for the direct comparison of persistence on hair, though for accurate analyses, hair samples weighing several grams are necessary. When trends only are required, hair samples of about one gram suffice. For cattle tick and buffalo fly work there is no standard test insect for the bioassay of clipped hair, and random samples taken from field collected populations of larval ticks and buffalo flies have to be used. For a discussion of this method and method (c) see Norris (1947).

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* An officer of the Division of Industrial Chemistry, seconded to the Division of Economic Entomology.

† In this paper, by DDT is meant: 2:2-bis(p–chlorophenyl)–1:1:1-trichloroethane.

2. Solar Radiation and Rain

DDT shows absorption in the region 2200–2500A (ultraviolet) (with a maximum at 2360A in 95 per cent. ethanol) and also in the infra-red but none in the region 2900–7000A (sunlight) when dissolved in carbon tetrachloride. This and the fact that DDT is a comparatively stable molecule, being decomposed easily only by alkalis, suggest that DDT would be stable under atmospheric conditions. Reports on the stability of DDT to sunlight and/or ultraviolet light have been some-what conflicting (Godkin and Swingle, 1945; Lindquist *et al.*, 1946) but when tested by biological methods it is generally agreed that the toxicity of DDT is reduced by exposure to these radiations. How-ever, Lindquist *et al.* report only a small reduction in toxicity to houseflies and they adduce evidence that decomposition, when it occurs, is restricted to the surface. Chemical estimations generally do not show a rapid loss of DDT under such circumstances which would be in accord with surface decomposition. The following experiments support this conclusion. As residues from emulsions might conceivably catalyse the decomposition, films formed from both solutions and emulsions were used.

Preliminary work had shown that exposing DDT to bright sunlight or unfiltered ultraviolet light (Mercura Lamp 83, 240 volts, 80 watts) for periods of a few days did not cause any detectable decomposition. Therefore, it seemed more practical to use an instrument which would give an " intensified sunlight." A twin carbon arc Weatherometer was used which gave a radiation closely resembling sunlight, it being only very slightly richer in the ultraviolet. Thin films of DDT (150 gamma DDT per sq. cm. in glass petri dishes), prepared from an alcoholic solution of DDT and from an emulsion* were exposed in the Weatherometer for 96 hours at 60°C., the humidity being relatively high as the base of the instrument consisted of a tank of water over which a steady upward flow of air was maintained. This period corresponds to an exposure for a period exceeding four months to the south-eastern Australian climate. Care was taken to see that all specimens received equal irradiation. The DDT was estimated by the dehydrochlorination method, after extraction with hot benzene, and only 56 per cent. of the pure DDT and 41 per cent. of the DDT prepared from the emulsion was recovered. Judging from the appearance of the films, it is possible that the high temperature caused loss of some DDT by evaporation. Despite the severe conditions of the experiment, this 50 per cent. loss of DDT, even had it occurred at ten times the rate, does not account for the recorded losses in the field. Moreover the films, after exposure, exhibited the normal toxicity of DDT to house flies. Fleck (1944) has shown that at 45°C. the rate of volatilization of DDT is very slow, and, under the experimental conditions, a 6·7 per cent. loss was recorded in 37 days.

' Formula:

DDT	.	2·0 g.
Solvent naphtha		12·5 ml.
(b.p. 90°–190°C.)		
Wetsit		1·25 ml.
Water to		1 litre

Further evidence to support this and to show the effect of rain is given by Norris (1947) who exposed some cattle hides to the sun and rain, others to the sun but protected from the rain, and found that although the exposed hides had lost an appreciable part of their toxicity after three and a half months, they still killed buffalo flies. In two recent papers (Peairs, 1946; Matthysse, 1946) importance is attached to both rainfall and sunlight to account for the loss in toxicity of DDT on cattle.

3. Growth and Loss of Hair

Cattle are considered to have one coat of hair annually, but there is no special shedding period, shedding taking place slowly throughout the year (Duerden and Whitnall, 1930). Longer fibres are grown in the winter and shorter fibres in the summer so that the coat is longest at the end of winter. There is thus an apparent post-winter moult, especially in the colder climates. Excluding this period, it is inconceivable that the natural shedding of hair would materially affect the persistence of DDT over a period of less than a month or two.

4. Skin Secretions

The rate of secretion of sebaceous material is generally slow as is shown for Australian Illawarra Shorthorn cows at Canberra (Table 1). Selected areas on two beasts were washed with alcohol and then with warm soapy water, hair samples being taken immediately before washing, six hours after washing when the hair had dried and then at weekly intervals for a period of three weeks. Sebaceous secretions were considered to be that material extractable with petroleum ether (b.p.40°–70°C.). The figures show that 35 per cent. (mean result) of the secretion present on the hair at equilibrium had been replaced in 21 days. Provided the secretion does not move outward along the hair then this indicates a 35 per cent. increase in the amount of hair present. Available information (cf. Duerden and Whitnall, 1930) suggests that such a large rate of increase in the amount of hair present is highly improbable. From the figures given for one animal by Duerden and Whitnall it can be calculated that the maximum average increase in the amount of hair over a three week period is approximately 15 per cent. and this occurred during the period of

TABLE 1.

Period	Mg Secretion per g Hair		
	Animal 1		Animal 2
Before washing	39·3	37 9	47 0
6 hours after washing .	3·7	0 0	4·6
7 days later .. .	6·0	6·4	8·2
14 days later . ..	12·2	10 8	10·1
21 days later • . .	18·0	13·2	20·4

rapid winter growth. Therefore the sebum must move along the hair in the direction of the tip. The validity of taking the pre-washing result as an equilibrium figure is shown in Fig. 2, where the curve for Series 1 shows a more or less constant value over the last 24 days of the experiment, this value being approximately equal to that at the commencement of the experiment.

· Even though the sebum moves outward along the hair and could consequently take the DDT with it, the results obtained from the following experiment clearly show that sebaceous secretions are not the most important factor in the loss of DDT.

5. Licking

Preliminary experiments had indicated that cattle do remove DDT from their hides by licking and it is significant that cattle, especially cows, are able to lick almost every part of the body save the head and neck. Moreover, cattle lick each other. At Yeerongpilly, Queensland, nine animals were sprayed as evenly as possible over an area of about two square feet on each side just behind the shoulder with an emulsion containing 2 per cent. DDT* using 500 ml. per beast. A mechanical atomizer was used, each area being sprayed to saturation. Before spraying three of the beasts the area to be sprayed was washed with alcohol and then with soap and water. These three and three more of the animals were prevented from licking themselves by fitting each beast with a piece of strong timber passing between the forelegs of the animal, the timber being kept in place by a headstall and surcingle (the latter being close to the forelegs). All animals were tethered in separate covered stalls during the entire experiment. The effects of direct sunlight were thereby eliminated. The tests were thus grouped in three series of three animals each:

1. Those in which no attempt was made to prevent the animals from licking the treated area.

2. Those in which precautions were taken to prevent the animals from licking the treated area.

3. Those in which precautions were taken to prevent the animals from licking the treated area which was washed before being sprayed.

An area of about one square foot was selected in the centre of each treated area and hair samples were taken from each side of each beast at the following intervals: before spraying, and 1, 3, 7, 15, and 31 days after spraying, and each hair sample was analysed for DDT and sebaceous secretion.

* Formula of emulsion concentrate:

DDT	12·8	per cent.
Benzene	15·8	per cent.
Dibutyl phthalate	18·7	per cent.
Wetsit	1·5	per cent.
Water	51·2	per cent.

For analysis the hair sample was extracted with petroleum ether (b.p. 40°–70°C.) which removed sebaceous secretions and DDT. The extract was filtered, evaporated to dryness, and the residue dried to constant weight at 100°C., it being established that this treatment did not bring about any decomposition of the DDT. The residue was dissolved in gracial acetic acid, filtered, and made up to an appropriate volume (50 ml. or 100 ml.). For the estimation of the DDT the colorimetric procedure of Chaikin (1946) based upon the intensity of colour produced when p–p′ DDT is heated in a mixture of glacial acetic acid and concentrated sulphuric acid was used, substituting a Lange photoelectric colorimeter with a suitable blue filter for the spectrophotometer.

The method was found to be reproducible, advantage being taken of the fact that the secretions were largely insoluble in cold glacial acetic acid. The error was estimated not to exceed 5 per cent. and the amount of sebaceous secretion was determined by difference. In all cases the clean hair was dried to constant weight and results expressed as mg. per g. clean dry hair. Expressing the results in this way enables the results from one hair sample to be compared with those from another since the results are then independent of the amount of moisture, dirt, sebum, DDT, etc. present in each hair sample.

The means and ranges of replicates for both DDT and secretion per g. hair are proportional, so by using logarithms the dispersion will be independent of the means. A statistical analysis was conducted using logarithms, and the antilogarithms or geometric means of the original values are presented in Tables 2 and 3, the logarithms being given in parenthesis.

TABLE 2.—TREATMENT MEANS, MG. DDT PER G. HAIR.

Days after Spraying				Series 1.	Series 2.	Series 3.
1 ..	..	..	..	13·7 (1·137)	24·1 (1·382)	7·9 (0·900)
3 ..	..	..	..	3·9 (0·588)	19·6 (1·292)	6·7 (0·827)
7 ..	..	..	..	..	12·7 (1·102)	5·0 (0·698)
15 ..	..	..	..	..	5·6 (0·747)	6·2 (0·793)
31 ..	..	..	..	..	6·3 (0·800)	9·2 (0·962)

Minimum difference for significance at 5 per cent. level for means of logarithms at the same date = 0·346.

Minimum difference for significance at 5 per cent. level for means between dates for the same treatment = 0·267.

TABLE 3.—TREATMENT MEANS, MG. SECRETION PER G. HAIR.

Days after Spraying.	Series 1.	Series 2.	Series 3
0 .	26·4 (1·422)	19·1 (1·282)	9·8 (0·992)
1..	74·5 (1·872)	93·8 (1·972)	31·1 (1·493)
3..	45·7 (1·660)	78·5 (1·895)	23·8 (1·377)
7..	20·7 (1·315)	62·8 (1·798)	16·1 (1·208)
15 .	23·0 (1·362)	45·5 (1·658)	38·3 (1·583)
31..	19·6 (1·292)	28·8 (1·460)	77·3 (1·888)

Minimum difference for significance at 5 per cent. level for means at the same date = 0·302.

Minimum difference for significance at 5 per cent. level for means between dates for the same treatment = 0·221.

On any one occasion a sample was taken from each side of a beast, the location of these two samples being balanced relative to one another about the corresponding centres of the areas subjected to sampling, these areas being rectangular in shape and symmetrically placed relative to the spine. The same sampling sites were cut on all beasts on all treatments at the same date. This procedure may have led to a confounding of gradients on the sampled area with time of cutting so that in making comparisons between different times of cutting, this factor should be recognized.

In Fig. 1 are recorded the amounts of DDT on the three series of animals in mg. DDT per g. hair, and in Fig. 2 the amounts of sebaceous secretions in mg. secretions per g. hair. It is interesting to note that in Series 1 no DDT was detected after seven days. Earlier work using a variety of emulsions had given a value of 1 mg. DDT per g. hair or less after a period varying from seven to ten days. After spraying there was an immediate increase in the sebaceous secretions but the amount per g. hair had returned approximately to its pre-treatment value after one week. The immediate increase in sebaceous secretions after spraying is presumably due to a stimulation of production by the emulsion used because it is known (Regan and Freeborn, 1936; Freeborn et al., 1934) that spraying cattle with petroleum oils or emulsions influences the physiological processes associated with the glands of the skin. The results given in Table 1 support this, for when cattle are not sprayed after washing no marked increase in secretions is recorded.

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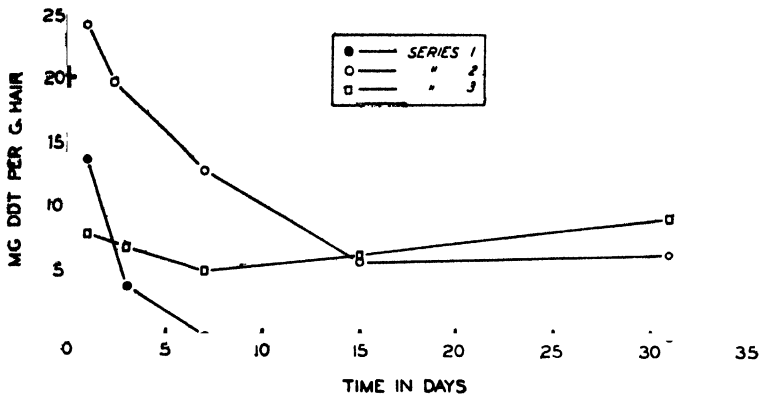
FIG. 1.—Amount of DDT on the hair of cattle in mg. per g. of hair.
Series 1: Cattle free to lick treated area.
Series 2: Cattle unable to lick treated area.
Series 3: Cattle unable to lick treated area which had been washed
before spraying.

Comparing Series 1 and 2, the differences three days after spraying, and at all subsequent samplings, are highly significant, and must be regarded as real. In particular, Series 2 appears to stabilize at about 6 mg. per gram of hair while no DDT residue is observable in Series 1. The lower value for Series 1 at one day after spraying bears the same relative relation to Series 2 although the difference is not statistically significant.
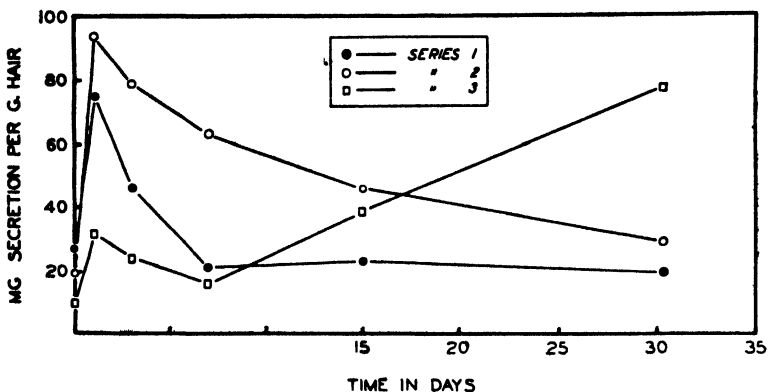
FIG. 2.—Amount of sebaceous secretions on the hair of cattle in mg. per g. hair.

As there was no difference in the treatment of the animals in Series 1 and 2 until after spraying, they all should have received a similar deposit of DDT at zero time. A reasonable extrapolation of the curves of both series (Fig. 1) is 30 mg. per gram of hair at zero time. Taking this value, the loss of DDT in Series 1 during the first day, is of the order of 50 per cent. Continuing at this rate the expected DDT residual after three days is about 4 mg., and after 7 days only 0·25 mg. per gram of hair. These calculated values are in accord with the actual values. This very rapid loss of DDT is not shown in either Series 2 or 3 and consequently licking, the only known variable, must be considered responsible. It was not possible to prevent the animals from rubbing which could have caused some loss but, as consideration of the results of Series 2 and 3 will show, this could not have been the major cause. Similarly the rapid loss of skin secretion produced immediately after spraying was due to licking. After seven days an equilibrium had been established and the animals were removing the secretion as rapidly as it was produced.

Series 2 consisted of animals which could not lick the treated area and there still remained on the animals 6·3 mg. DDT per g. hair at the end of the experimental period (31 days). The amount of DDT present dropped from a value of 24·1 mg. DDT per g. hair one day after spraying to an approximately constant and apparent equilibrium value after 15 days, the results not being significantly different from those for Series 3. Such a rapid initial fall can be accounted for only by rubbing, induced by irritation, removing flakes of the epithelium and skin secretions. A similar trend is shown for the skin secretions in Fig. 2. As in Series 1, spraying has induced a rapid initial increase in the amount of secretions on the hair which is followed by a rather rapid loss which is consistent with rubbing having removed the secretions. Even with this loss, the amount of secretions on the hair after 31 days was greater than that present before spraying.

Series 3 shows a somewhat clearer picture. The amount of DDT on the hair remained approximately constant throughout the entire experimental period, the observed variations not being significant. Washing removed the major part of the sebaceous secretions prior to spraying which left the hair considerably cleaner than that of the animals in Series 2. Consequently, less DDT, i.e. less emulsion, was retained by the hair of these animals than by the animals of Series 2. The amount retained, however, appears to be an equilibrium amount for the emulsion used since approximately the same amount was observed on the hair of animals of Series 2 after 15 days. As before, an increase in sebaceous secretions was recorded one day after spraying. Washing would remove much of the secretions from the skin and some of the secretions produced immediately after spraying would be used to replace this, thus giving a lower value after one day than that given by Series 1 or 2. There followed a slight decrease and then a continuous increase due, no doubt, to the accumulation of secretions, because the cattle were unable to remove them by licking. The reason for the decrease between one and seven days could be the same as that advanced in Series 2, viz. removal by rubbing which is in accordance with the slight decrease in the amount of DDT recorded after the first day. Even though irritation may lessen after one week

stimulation of production may still be present owing to the oil having come into contact more intimately with the skin. Consequently the amount of secretion present after 31 days (viz. 77·3 mg. per g. hair) may be abnormally high.

Since it is evident that cattle remove much of the DDT applied to their skin and hair by licking, the possibility of the DDT proving toxic to cattle becomes important. Of all domestic animals, cattle appear to be the most susceptible to DDT (Plummer, 1946; Orr and Mott, 1945). From the results of spray tests, it is unlikely that an animal weighing 500 kg. would retain on the hair and skin more than 50 g. DDT when sprayed (or dipped) over the entire body with an emulsion containing 2 per cent. DDT, i.e. 100 mg. per kg. of body weight. Assuming this to be removed uniformly over a period of seven days, there would be ingested daily approximately 14 mg. per kg. body weight, which from published data for the oral administration of DDT as a powder. would appear to be harmless. Added to this, cattle in warmer climates carry less hair than those in the colder climates and so would retain less DDT, and the tendency now is to use less concentrated LLT preparations. For the animals used in the experiments it is estimated that the amount of DDT retained after spraying, assuming the whole animal to have been sprayed, was about 50 mg. per kg. of body weight which, when removed in seven days by licking would correspond to the ingestion of 7 mg. DDT per kg. of body weight daily. No symptoms of DDT poisoning were observed. Plummer (1946) reports that cattle sprayed over the entire body with a preparation containing 5 per cent. DDT remained normal. Orr and Mott (1945) have shown that the daily oral administration of 100 to 200 mg. DDT per kg. of body weight as a dry powder to cattle for a period of three weeks is not acutely toxic and that most of the material is eliminated from the body, although small quantities may be taken up by the blood stream. Telford and Guthrie (1946) secured no definite evidence to indicate that any harmful effects resulted from spraying a Holstein cow with a preparation containing 10 per cent. DDT.

Stimulation of secretion production would, no doubt, increase the amount of licking which would remove the DDT more rapidly. Moreover, it seems reasonable to assume that the old secretion carrying the DDT, either in solution or suspension, is pushed towards the end of the hair by new secretion and since the old secretion is removed first, the DDT is lost. If this stimulation of secretion production could be prevented then the period of persistence may well be increased. This may be possible if an aqueous instead of an oil carrier is used for the DDT, e.g. a water suspension. The advisability of adding to a DDT emulsion a substance to make it so unpalatable to the cattle that they would refrain from licking is doubtful because the cessation of licking may be injurious to their health.

6. Absorption into the Hair

Absorption into the hair has been advanced as a factor to account for the loss in toxicity of DDT on cattle. Chemical analyses depend on extraction of the hair samples with a solvent to remove the DDT; such treatment must recover any DDT actually present inside the

hair. The DDT analyses, obtained in the previous experiment, eliminate absorption into the hair as a factor of any importance contributing to the loss in toxicity of the DDT. No sorption of DDT, by cattle hair, from DDT solutions (alcohol and toluene) could be detected. Cattle hair removed, by sorption, small amounts of DDT from emulsions containing 2 per cent. DDT (3 mg. per g. hair; about 3 g. per beast). Little is known about absorption into the skin, but the work described above shows that such a process, if it does occur, cannot be of importance in removing DDT from the hair (cf. Series 2 and 3).

7. Conclusion

From the foregoing work, it is concluded that in the field the major cause of the removal of DDT from the hair of cattle sprayed with DDT preparations is licking, eitner by the animal concerned, or by another animal. All the other factors discussed play a very minor role and of these factors the effects of rubbing, solar radiation, and the leaching action of rain would appear to be the most important. In earlier work it soon became evident that the composition of the emulsion had little effect on the period of persistence and that increasing the DDT concentration from 1 to 2 per cent. produced but a very small increase in the persistence period. These observations are readily explained when it is realized that it is the animals themselves which remove the DDT. These results also suggest that adequate control of ticks and flies as distinct from eradication, would be obtained by preparations containing less than 1 per cent. DDT.

8. Acknowledgments

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The Etiology of Take-all Disease of Wheat

3. Factors Concerned with the Development of Take-all Symptoms in Wheat

By N. H. White, D.Sc.*

Summary.

1. In a glasshouse experiment using soil from take-all and " healthy " areas, take-all symptoms developed in wheat when the soil was not steam-sterilized and when steam-sterilized but inoculated with *O. graminis*.

2. There was no difference in the amount of take-all when the wheat was grown in soils taken from these two field localities. It was suggested that soil differences, if they existed, did not influence the pathogenicity of *O. graminis* on wheat.

3. In wheat grown in steam-sterilized soil from either of these two localities no take-all symptoms occurred.

4. In steam-sterilized soil inoculated with *O. graminis* and a dry top soil maintained throughout the growing period, take-all symptoms developed in all plants.

5. In this experiment whiteheads were developed by all plants producing ears.

6. Examination of white-eared and normal-eared plants taken from the field during three seasons revealed that there was significant proportionality between the functioning roots and the load of the shoot system.

7. The relationship of the functioning roots to the shoot load was discussed with reference to the development of the whitehead condition and the collapse of the plant.

In the second part of this series of studies it was shown that *Ophiobolus graminis* appeared to be the primary invading organism of the roots and crowns in take-all affected wheat in the field. Also that the distinction between the condition of the roots and crowns of white-eared and normal-eared plants in the field appeared to be one of degree only. There remained the need to establish the pathogenicity of *O. graminis* on wheat grown in soil from the field under observation, and to account for the conditions which determined whether a plant became a white-eared or normal-eared plant, once affected with *O. graminis*.

1. The Pathogenicity of *O. graminis* on Wheat in Sterilized and Unsterilized Soil from Take-all and Healthy Areas of the Same Field

Isolates of *O. graminis* obtained from affected plants in the field reported in the first part of this series, were submitted to the usual seedling pathogenicity test in pots under glasshouse conditions. The isolates showed variability in pathogenicity ranging from mildly to severely pathogenic. A moderately pathogenic strain was used in the following investigations.

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In the first part of these studies it was stated that the location of a take-all patch may be due to soil differences or to the concentration of *O. graminis* inoculum in localized areas. Therefore for the testing of the pathogenicity of *O. graminis* on wheat, soil from the site of take-all patches and healthy areas was used in order to determine whether the location of the soil affected the severity of the symptoms when the same isolate of the fungus was used.

The pathogenicity test was carried out under glasshouse conditions so that some degree of control of environment might be obtained, and was conducted in the 1940 season.

Soil taken from the top six inches was collected from some of the areas known to be badly affected with take-all in the 1939 season, and a similar lot of soil was collected from adjacent areas that produced healthy plants. The soil from these two areas was air-dried, and sieved through half-inch mesh wire-netting to remove stones. No attempt was made to exclude diseased plant material.

To each of 80 Wisconsin temperature tank cans eight kilograms of soil were added, 40 cans contained soil from take-all areas and 40 cans from healthy areas. Twenty of these cans containing soil from each of the areas were steam-heated for six hours. Paper was tied over the tops of these and they were allowed to stand for three weeks before commencing the experiment. There were in all, four lots of soil each contained in twenty cans. They were take-all area soil steam-heated, take-all area soil unsterilized, healthy area soil steam-heated, and healthy area soil unsterilized. The soil in ten cans of each of these four lots of soil was inoculated at seeding with straw inoculum of a moderately pathogenic strain of *O. graminis*.

Before seeding with wheat, the soil in the cans was moistened to about field capacity. Inoculum was so placed that it was 2 inches below the surface, and surface-sterilized " Bencubbin " wheat seed was placed half an inch above the inoculum. Eight seeds were sown in each can and the cans were randomized. Water was not added until the seedlings emerged and from then on the soil was kept moist by adding water to the top of the soil as required. However, no attempt was made to control the soil moisture at a definite level, but every precaution was taken to avoid creating conditions of drought or waterlogging.

Four weeks from the commencement of the experiment the number of plants in each can was reduced to four. At heading-out stage, two cans were removed from each of the four treatments containing soil from take-all areas, and one can was removed from each of the four treatments containing soil from healthy areas. The plants in these cans were dug up and the roots examined for lesions. Many of the lesioned roots were plated to ascertain their microfloral content.

Plants in unsterilized soil (whether inoculated or not) and steam-sterilized inoculated soil had lesions on about 75 per cent. of their crown roots, and the seminal root system on nearly all these had completely rotted away. No lesioned roots were found on the plants from the steam-sterilized uninoculated soil. Plating of the lesioned roots from each treatment gave the usual types of fungi found at heading-out in lesioned wheat roots, including species of *Fusarium*,

Penicillium sp., *Helminthosporium sativum*, and *Curvularia* sp., reported in Part 2. However, *O. graminis* was obtained in about 20 per cent. of the roots plated.

Results.

In each of the treatments data were obtained on (i) the percentage of seedling blight, (ii) the amount of tillering at jointing, (iii) the number of tillers producing ears, (iv) the yield of grain, and (v) the percentage of take-all in the mature plants. A summary of these data are given in Table 1. As a result of analysis of the data from all the treatments, there was no significant difference between soil types, i.e. take-all area soil, as against healthy area soil, whether inoculated or not. For all measures of pathogenicity the difference between inoculated and uninoculated sterilized soil was highly significant. For unsterilized soil there were significant differences between the uninoculated and inoculated series only in the percentage of plants with seedling blight and the number of tillers per plant.

Take-all symptoms as manifested at maturity varied from plants that died prematurely at heading-out either before or after the ears had emerged (white ears), to plants that were hard to distinguish from the normal ripe plants but yielded very shrivelled grain. To a large extent this obtains under natural field conditions, but owing to the artificial conditions of the greenhouse the distinction between a mature ripened plant and a prematurely ripened plant was even less apparent; this was due largely to the fact that diseased plants did not show the stunted growth as seen in the field, and ripening was more gradual in the greenhouse. Upon harvesting the criterion of a take-all plant was one that produced an average grain weight of less than 0·053 g. per grain. This standard was based on the weight per grain of known take-all plants collected in the field.

The data revealed that take-all occurred in all treatments except in the sterilized uninoculated soils from both take-all and healthy areas.

Steam-heating of the soil apparently had a twofold effect.

(i) The pathogen already in the soil was killed, as evidenced by the absence of symptoms of disease and root lesions on plants grown in sterilized soil in contrast to the symptoms and root lesions on plants grown in sterilized inoculated soil.

(ii) Increased vigour of the wheat plants, manifested in density and greenness of foliage, early maturity, height of the plants, number of tillers attaining maturity, and total yield of the plants seen in Fig. 1. This phenomenon resulting from steam-heating of soils of different types was noted before by the writer and there are frequent references to it in the literature. Johnson (1919) has summarized the theories explaining this, and the results of his investigations suggest that the beneficial action of heating soils is due largely to the liberation of ammonia.

Potter and Snyder (1918) found that heating the soil " caused an increase in soluble non-protein nitrogen." Piper (1942), however, found that the availability of copper was significantly increased by partial heat-sterilization of copper-deficient soil, and suggested that the increased availability of copper was either a direct result of heat,

or by the elimination of competition from some soil microflora and fauna. Millikan (1941) claims that the beneficial effect of steam-sterilization of Wimmera soils is due to the removal of soil inhabiting organisms that compete with the plant for soil nutrients and states that " formalin-sterilization induced a similar or better improvement of growth to steam-sterilization." In the writer's experiment, the occurrence of seedling blight and the white-head condition in plants grown in the steam-sterilized soil inoculated with *O. graminis* suggests that this fungus is able to attack the roots of the wheat plant in spite of the enhanced nutritional benefits to the plant through steam-sterilization of the soil.

FIG. 1 —The effect of steam-sterilizing soil on the growth of wheat. From left to right: Steam-sterilized soil from take-all area, steam-sterilized soil from healthy area, unsterilized soil from take-all area, unsterilized soil from healthy area.

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2. The Effect of Dry Top Soil on the Development of Take-all Symptoms

Field observations indicated that a relatively dry top soil was an important contributing factor in the development of take-all symptoms in the wheat plants when the seminal root system had been destroyed by parasitic organisms. The greater frequency of the white-head condition in cans with a relatively dry top soil compared with those containing moist top soil confirms field observations on comparable soil conditions. An experiment was therefore undertaken in 1941 to determine the effect of a dry top soil on the development of take-all symptoms. Soil collected from the top 6 inches of a fallow area in the naturally take-all affected field was air-dried, sieved, and steam-heated for six hours and allowed to stand several weeks before the experiment was commenced. The soil was found to have a field capacity of 18 per cent., and a permanent wilting coefficient of $6 \cdot 8$ per cent. of the dry weight.

Eight kg. of the air-dried steam-sterilized soil were weighed in each of 40 Wisconsin temperature tank cans. The soil in twenty cans was brought to field capacity by adding the requisite amount of water, and the soil in the remaining twenty cans was brought to 65 per cent. of the field capacity by thoroughly mixing in the necessary amount of water. Two L-shaped glass irrigators, 1 cm. in diameter, were so placed in the soil in each can, that the outlet of one of them was one-third and the outlet of the other two-thirds of the distance from the bottom of the soil to the top of the soil. Straw inoculum of a six-weeks-old culture of *O. graminis* was placed in ten cans of each of the dry and moist series, at a depth of 2 inches below the surface. Three surface-sterilized and germinated (two days) wheat seeds were placed in each can at a depth of 1½ in. (The seed used in this experiment was from a single plant and the progeny of three generations of selfed " Bencubbin " wheat.) The water lost from the soil in each of the cans was replaced by adding enough water through irrigators to bring the cans back to their original weight. During the first two months of the experiment this was done only once a week, but after that and until the plants were ripening, every three days. Although there were fluctuations in moisture content of the soil below the two initial standards, one series was maintained in a *wet* condition throughout, while the other series was kept in a moist condition only in the lower part of the soil, the top 1½ inch gradually drying out simulating the condition frequently observed in the field.

Results.

By inspection, differences between each of the treatments were quite obvious long before the plants reached maturity, as shown in Fig. 2.

By the time the experiment was completed, differences appeared even greater, as shown in Fig. 3. Quantitative data when analysed proved that these differences were significant. A summary of the data is given in Table 2.

It will be seen that take-all disease occurred in soils wet throughout and in soils partially wet, when inoculated with *O. graminis*; but all plants in the series with a dry top soil were diseased plants, whereas only 54 per cent. of the plants in the series with wet soil throughout

showed symptoms of disease in the shoots. Furthermore, in the dry top soil series the symptoms were indistinguishable from those found in the field, which vary from plants killed in the seedling, jointing, and in-the-boot stages as well as at anthesis when the white-head condition is observed. All these stages, together with the stunting effect, were obtained in the dry top soil series as is illustrated in Fig. 3. On the other hand, symptoms shown by plants in the inoculated wet-soil series were atypical, and the plants gave the usual delayed maturity following seedling blight, seen in Fig. 2.

FIG. 2.—The effect of growing wheat in steam-sterilized soil with a dry and wet top soil From left to right: Dry top soil control, wet top soil control, dry top soil inoculated, wet top soil inoculated. (Each treatment in two rows)

The inoculated treatments in both the soil moisture series are significantly different from the controls in all the data shown in Table 2. This conforms with previous observations and the result obtained in the previous experiment. The introduction of *O. graminis* into the soil seriously affects the growth of wheat plants as well as causing the development of take-all symptoms.

The difference between the control series in the dry top soil as compared with the control series in the wet top soil is seen most conspicuously in the height and number of tillers as well as in yield and number of crown roots per plant (see Figs. 2 and 3). The significantly smaller growth of the plants in the dry soil series is explained by the findings of Briggs and Shantz (1913), Shantz (1927), Veihmeyer (1927), and Martin (1940). They have shown that it is impossible to maintain a mass of soil at a uniform moisture content below field capacity, and that the addition of less water than required to bring the soil to field capacity would result in some of the soil

TABLE 2.—SUMMARY OF DATA ON EFFECT OF DRY TOP SOIL ON THE INCIDENCE OF TAKE-ALL, AND GROWTH OF WHEAT IN SOIL FROM A TAKE-ALL AFFECTED FIELD.

Initial Moisture Condition of Soil.	Treatment.	Percentage of Take-all.	Mean Number of Tillers per Plant.	Mean Height per Plant	Weight of Grain per Plant.	*Grain Rating per Plant.	Mean Number of Crown Roots per Plant.	Mean Number of Non-lesioned Crown Roots per Plant.
				cm.	g.			
Field capacity	Control	0	7·9	99·6	9 8	9·8	60 3	60·3
	Inoculated	54	8 9	74·3	4 1	7·1	27·9	6 5
Field capacity, 65 per cent	Control	0	5·7	86·1	6 5	9 6	46·4	46·4
	Inoculated	100	2 8	40 3	4·3	4·3	37 0	11·1
Control compared with inoculated	Mean difference	..	3 5 xxx	35·5 xxx	4 0 xxx	4·0 xxx	20·9 xx	44·5 xxx
Field capacity compared with 65 per cent field capacity	Mean difference	.	1·6 x	23 7 xxx	1 5	1·5 x	2·4	4 6

Level of significance —xxx P < 0·001, xx P < 0·01; x P < 0·05.

* Grain rating refers to fourteen grades of grain size ranging from 0·000 to 0·070 per grain, with a uniform interval of 0·005 g. per grade. Ratings of 0·7 are equivalent to the rating of take-all plants in the field.

remaining unchanged so that plants growing in soil with an initial moisture content below field capacity would be forced to grow in a smaller volume of soil. Therefore in the series commenced at 65 per cent. field capacity the plants were forced to grow in a smaller volume of soil than those growing in the soil with an initial soil moisture at field capacity. In the dry top soil series then, the lower 65 per cent. of the soil was brought to field capacity at each weighing and it was this amount of soil that the roots had to draw on for plant nutrients. Plants in the inoculated soil commenced at 65 per cent. of field capacity simulated the field appearance when dry conditions prevail at any stage during their development if their roots were partly destroyed by the fungus. Under these circumstances when the top soil dried out, the new crown roots developing in this zone were unable to obtain sufficient moisture to maintain the minimal water requirement of the plants. On the other hand, in the soil brought back to field capacity at each weighing the top soil was always moist and new crown roots, whose development is always stimulated by root destruction, were able to replace the destroyed roots and maintain the water supply to the plants. However, the limiting effect on growth of the plants through root destruction by O. graminis is readily seen in the inoculated wet top soil series when compared with the controls (see Fig. 2).

3. The Relation of the Root to the Shoot System in White-eared and Normal-eared Plants

In an attempt to account for the conditions which determine whether a plant becomes a white-eared or normal-eared plant, the relative amount of shoot tissue to the number of surviving roots in both kinds of plants was critically examined during three seasons.

FIG. 3.—The effect of growing wheat with a dry and a wet top soil as seen when the plants are ripe. From left to right as in Fig. 2. It will be noted that the plants in the dry top soil inoculated can are typical take-all affected plants.

Method.

Samples of white-eared and normal-eared plants were collected in each of the three seasons 1940, 1941, and 1942 on plots sown continuously to wheat, and in 1942 on a plot sown to wheat for the second time after one year's fallow and a plot sown to lucerne in the previous year.

The samples were collected each year at the critical period, that is between anthesis and ripening, when it was easy to distinguish the white-eared plants from the intermediate slate-green plants; which later produced pinched grain, and the normal green-eared plants bearing plump grain. Only plants with fully emerged ears were collected, so that a very large proportion of white-eared plants which had one or more ears in the boot at the collapse stage were passed over. This procedure tended to restrict the white-eared samples to plants which collapsed latest in the season and which therefore should have been least affected of white-head samples by root destruction after collapse. The fact that plants in the selected white-eared sample were clearly distinguished from less and more severely affected plants probably left less room for variability than with normal plants which may range from just better than the intermediate slate-green to complete freedom from fungal attack. The plants were collected on an ear class basis and in order to secure a sufficient number in each ear class up to four ears, a rough tally was kept of the number collected in each class, and the white-eared plants were chosen so as to keep the numbers roughly equal. Whenever a white-eared plant was pulled, the near neighbourhood was searched for one or two normal plants of the same ear class. This was usually achieved within 2 feet of the white-eared plant.* In 1942 the procedure had to be reversed where, on finding a normal-eared plant, the neighbourhood was searched for a matching white-eared plant. In all instances the selection of the plant was determined solely by the shoot condition. In each plot all samples were drawn from an area not exceeding 200 feet by 50 feet.

Most of the plants collected had earless tillers 6 to 9 inches high which were dead before the ripening stage. The undeveloped tillers apparently are of some importance, however, because the number of crown roots for plants in an ear class increased in proportion to the number of partially developed tillers. Therefore in tabulating the data it was necessary to prepare two-way tables so that each plant could be classified according to the number of eared tillers and partially developed tillers.

The plants were examined in detail, and data on the following were obtained in 1940 and 1941: (i) Number of eared tillers; (ii) number of partially developed tillers; (iii) length of each eared tiller (length of last internode in 1941); (iv) total number of spikelets per ear; (v) total number of crown roots and number of crown roots with

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* When examining the plants in the laboratory many plants with high tiller number proved to be two or more inter-locked plants so that the numbers finally recorded were greatest for one-eared plants.

lesions;* (vi) total number of seminal roots and number of seminal roots with lesions; (vii) condition of sub-crown internode (lesions or no lesions). In 1942 the length of the eared tillers and the total number of spikelets per ear were omitted.

Results.

Because it would be impracticable to present the two-way tables for the three seasons for each particular and since the mean number of undeveloped tillers (1 to 1·5 per plant) is about the same for both white-eared and normal-eared plants of the same ear class, only the averages of all plants of the same ear class are presented. These are given in Table 3 which shows the mean values for each ear class for normal and white-eared plants in 1940, 1941, and 1942.

The results for the first two seasons differ markedly from the 1942 season in the data on the number of surviving crown and seminal roots and in the percentage of sub-crown internodes and first internodes with lesions. These differences appear to be related to the much higher rainfall of this last season (see Table 4), when 2,148 points fell between May and November inclusive in contrast to 626 and 899 points for 1940 and 1941 respectively. The data convey the general impression that with increasing moisture in the spring there is a decline in the number of surviving roots required to carry the white-head plants up to the point of collapse at the standard condition of fully emerged ears.

In 1940 the difference in the numbers of surviving crown roots for white-eared and normal-eared plants was small, so that for this season the number of surviving seminal roots must have been all-important. At the writer's suggestion root amputation experiments were carried out during this season in this field by Ludbrook (1942). It was found that severing the sub-crown internodes affected the plants more severely at all stages of growth investigated than did amputation of the crown roots, and thus provided supporting evidence of the importance of the seminal root system during this season (see Table 3). In 1941 the difference in surviving crown roots of white-eared and normal-eared plants was greater. This suggests that the crown root system was more effective in maintaining the plant in the later stages of growth than in 1940, and this again appears to be related to the spring rainfall which was slightly better in the 1941 season (see Table 4). In both seasons the number of surviving crown roots of white-eared plants increased with the number of ears produced by the plant, while the mean number of surviving seminal roots was about constant.

In 1942 practically all the crown and seminal roots of the white-eared plants had lesions. The moist conditions of the soil probably meant that only a few functioning roots were necessary to sustain the plant to the point of collapse and plants with more functioning roots would come into the intermediate class. The contrast between the number of functioning roots in this and previous seasons may have been exaggerated by much rotting of roots in the moist soil subsequent to collapse but prior to examination.

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* The criterion adopted for designating a root with lesions was: A root showing marked dark brown to black discolouration of tissue completely encircling the root for a variable distance and which in breaking revealed a blackened or discoloured stele usually rendered brittle through necrosis.

TABLE 3 (PART I.).

Ear Class.		Number of Plants.			Total Crown Roots.			Lesioned Crown Roots.			Surviving Crown Roots.		
		1940.	1941.	1942.	1940.	1941.	1942.	1940.	1941.	1942.	1940.	1941.	1942.
1	Normal ..	65	53	34	15·8	16·7	13·2	1·7	5·3	8·0	14·1	11·4	5·2
	White ..	40	73	78	11·9	13·5	11·7	7·6	8·6	9·9	4·3	4·9	1·8
2	Normal ..	55	42	26	20·1	26·0	20·0	3·3	7·4	1·5	16·8	18·6	18·5
	White ..	36	56	27	27·0	23·8	22·1	12·0	12·7	20·2	15·0	11·1	1·9
3	Normal ..	57	33	27	31·8	35 4	30·1	4·0	8·6	3·8	27·8	26·8	26·3
	White ..	30	42	30	26·1	32·1	23·2	14·1	19·3	20·1	22·0	12·8	3·1
4	Normal ..	12	18	27	38·7	42·3	39·2	4·0	10·4	7·0	34·7	31·9	31·9
	White ..	10	14	25	43·2	45·3	37·5	14·1	23·1	37·0	29·1	22·2	·5

TABLE 3 (PART II.).

Ear Class.		Surviving Seminal Roots			Percentage Sub-crown Internodes without Lesions.			Percentage First Internodes without Lesions		
		1940.	1941.	1942.	1940.	1941.	1942.	1940.	1941.	1942.
1	Normal ..	3·0	3·0	1·1	91	66	9	97	98	71
	White ..	·5	·7	·1	25	18	5	80	67	22
2	Normal ..	3·5	3·3	4·5	87	64	77	98	100	84
	White ..	·5	·6	·1	28	27	0	69	84	15
3	Normal ..	3·9	4·5	3·4	82	82	67	93	91	88
	White ..	·7	·9	·2	40	29	3	93	31	20
4	Normal ..	4·4	3·7	3·2	100	78	48	100	89	66
	White ..	·6	·7	·0	20	14	0	50	43	0

It would be appropriate at this point to note the trends apparent within the ear classes in the 1940, 1941 data (shown in Table 3). (a) White-eared plants—the total number of crown roots increased at the rate of 3·3 per undeveloped tiller but there was only a slight increase in non-lesioned crown roots per plant. There was no corresponding tendency for surviving seminal roots per plant to increase or decrease. There was no apparent trend in the percentage of plants with sub-crown lesions. (b) Normal-eared plants—the total number of crown roots increased at the rate of 3·5 per undeveloped tiller, there being no tendency for the number of roots with lesions to increase or decrease. For other data the trends were similar to those for the white-eared plants. In 1942 the tendencies in the crown and seminal roots were the same as for the previous seasons except that there was no evidence of any trend in the very small numbers of surviving crown roots.

TABLE 4.—RAINFALL IN POINTS AT CANBERRA DURING 1940, 1941, AND 1942, WITH STAGE CROP DEVELOPMENT

Month	1940	1941	1942	Stage of Crop
January	185	674	22	Stubble
February	10	257	131	
March	6	· 148	151	
April	696	54	7	Sown
May	115	186	638	Seedling
June	52	114	338	Seedling
July	29	48	194	Early jointing
August	91	105	229	Jointing
September	244	262	244	Late tillering
October	25	124	174	In boot
November	70	90	431	Anthesis and dough
December	177	96	112	Ripe (harvested)

It was hoped that it would be possible to show that if a seminal root was rated as effective as k crown roots, the value of k to depend on the season, then the total effective root system of white-eared plants would be proportional to the number of ears. Values of 12 and 2 for k best satisfy the data for 1940 and 1941 respectively. This approach is of interest but cannot be regarded as entirely satisfactory owing to its sensitivity to small systematic errors in assessment of root lesions.

From these data it would seem that the seasons in 1940 and to a less extent in 1941 were conducive to the development of the so-called "dry weather take-all" (Russell, 1934) and data for these seasons

provided evidence of a relationship of the shoot system to an effective absorbing root system in white-eared plants. In 1942 a relation of this sort was not apparent but it may have existed at the initial development of white ears. Subsequent rotting of the few roots per ear functioning at collapse would destroy the evidence of proportionality.

4. Discussion

The pathogenicity of *Ophiobolus graminis* in relation to take-all disease has been established by many investigators and is reviewed by Garrett (1942). Past workers have used the effect of infection on seedling plants and on yield as criteria for the pathogenicity of *O. graminis* on wheat. This has proved useful in assessing the capacity of the fungus to attack root tissue, but does not show the relation of the fungus to the final stage of take-all disease manifested as white-eared plants known as "whiteheads," and this is the dominant field symptom. The experimental development of the whitehead condition is claimed by a few workers only (Geach, 1932; Garrett, 1941; White, 1942; and Angell, 1943). However, the experience of all was that only some of the plants became typical white-head or white-eared plants at maturity. This agrees with the field conditions in so far as white-eared plants occur scattered throughout the crop, but the significant and characteristic feature of take-all is that it occurs in patches where only few plants escape. In the studies reported here the writer attempted to reproduce experimentally the whole range of symptoms, particularly the whitehead condition, in all the plants and in every replication of the treatment.

It was first necessary to eliminate the soil factor alone as a possible cause of the symptoms. The pathogenicity experiments in soils from both take-all and healthy areas demonstrated that the presence of *O. graminis* was necessary for the development of take-all symptoms in plants, and that soil taken from either take-all or healthy sites had no effect on the pathogenicity of the fungus. However, what appeared more important was soil condition as demonstrated by the higher incidence of the disease in cans with dry top soil. This was confirmed in a subsequent experiment where a dry top soil was maintained throughout the growing period of the plant.

In this experiment it was found that all plants in each of the replications with a dry top soil developed symptoms when inoculated with *O. graminis* and all plants that produced ears became whiteheads without exception. These plants were indistinguishable from those affected with take-all in the field; the plants in one of these cans are shown in Fig. 3.

The complete development of take-all in this treatment leaves little room for doubt that at least two primary conditions for the development of the disease are the presence of *O. graminis* and a dry top soil. These conditions would simulate those for the "dry weather take-all" (Russell, 1934), and were those obtaining in the field under observation during the 1940 and 1941 season when studies were made on the ratio of shoot to functioning roots.

The collapse of the plants at any stage of development, but particularly at maturity, might be explained on two grounds, namely, destruction of the roots, and the production of toxic substances by the fungus.

If the production of toxins was responsible for the collapse of the plant, as in vascular wilt diseases, a marked disparity would not be expected to occur between the number of lesioned crown roots on plant, as in vascular wilt diseases, a marked disparity would not during the three seasons reveal that there was a very marked disparity.

Evidence in favour of root destruction would be proportionality in effectiveness of the roots and the load of the shoot system at collapse for plants with different loads. The results for the 1940 and 1941 seasons may be interpreted this way. In the wet spring of 1942, rapid destruction of root tissue subsequent to collapse but before examination, may account for the failure of the season to support the hypothesis. The data in Table 3 show that at maturity all plants had roots with lesions and it was shown elsewhere (White, 1945) that only a percentage of these become white-eared plants. From the writer's findings it seems that whether a plant will be whitehead or normal-eared depends on the capacity of the roots to function above a certain threshold value. This value is affected by the load of the shoot system on the functioning roots and by the moisture available in the soil horizons. The root amputation experiments of Simmonds and Sallans (1929, 1933), and Ludbrook (1942), showed that either seminal or crown roots or both could function in absorbing water from the soil. With the destruction of the seminal roots the crown roots function. When the top soil is dry the surviving crown roots are unable to function and "dry weather take-all" develops as was the case in the writer's experiment. When the top soil is wet then the crown roots could function above the threshold value, provided there were enough of them. Should their development be impaired through the destruction of the root primordia in the crown and first node by *O. graminis* or their rate of destruction exceed their development, then in spite of the wet top soil the functional value of the crown root system will fall below that of the threshold value of the plant concerned with a particular shoot "load." Such a plant will collapse and develop the white-eared condition.

5. Acknowledgment

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The Etiology of Take-all Disease of Wheat

4. The Effect of Agronomic Practices on the Incidence and Severity of Take-all

By N. H. White, D.Sc.[*]

Summary.

The effect of different agronomic practices on the incidence and severity of take-all disease was investigated in a naturally affected field at Canberra.

It was found that two years' bare fallow resulted in the eradication of *O. graminis* from the soil, as indicated by the complete absence of take-all and freedom from root lesions. Cropping to wheat for four years increased the severity of the disease and decreased crop vigour.

One year's fallow or oats in rotation with wheat, or oats-fallow-wheat rotation decreased the severity of take-all and increased the yield.

General conclusions on the etiology of take-all disease are given.

1. Plan of Investigations

In the previous parts of the study on the etiology of take-all disease in a naturally affected field at Canberra, it was shown that there was a causal relation between *Ophiobolus graminis* Sacc. and the development of take-all symptoms. In order to obtain complementary information on the incidence and severity of the disease different agronomic practices were carried out on the 2-acre block in this field for a period of three years.

The choice of the various treatments was influenced by the reported facts concerning the disease. It has been shown repeatedly that control may be obtained by a one-year break from wheat under oats, any non-cereal crop excluding pastures, or bare fallow. Also that continuous cropping to wheat usually increases the severity of the disease, though some exceptions have been reported (Russell, 1934).

In 1940 the 2-acre area was surveyed into eight equal blocks, each measuring 200 ft. by 50 ft. The following treatments and rotations were applied to each of these blocks respectively:—(i) Wheat followed by fallow, (ii) two years' pasture (ryegrass–subterranean clover mixture), (iii) two years' bare fallow, (iv) two years' lucerne, (v) and (vi) continuously cropped to wheat, (vii) oats followed by fallow, (viii) wheat followed by oats. In 1939 the area was under wheat and the disease occurred in patches over the whole area, as shown in Fig. 2 in Part 1 of this series (White, 1945). In the 1942 season wheat was grown on all the blocks, and data on the following was obtained from each block:—(i) Percentage of take-all, (ii) grain yield per ear, (iii) yield in bushels per acre, (iv) mean height per plant, and (v) mean number of tillers per plant. To determine the percentage of take-all and the numbers of tillers per plant, specimens were examined in 18-in. square quadrats, spaced at staggered intervals in twenty locations in each plot. For determining grain yield per ear, 500 ears were collected at random by walking along five transects spaced equally apart in each block. In each block random samples of plants were taken for examination for root lesions.

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TABLE 1.—THE EFFECT OF AGRONOMIC PRACTICES ON THE INCIDENCE AND SEVERITY OF TAKE-ALL ON WHEAT AT CANBERRA IN THE 1942 CROP.

Plot Number.	1.	2.	3.	4.	5.	6.	7.	8.
Treatments—1940 ..	Wheat ..	Pasture ..	Bare fallow	Lucerne ..	Wheat ..	Wheat ..	Oats ..	Wheat
1941 ..	Fallow ..	Pasture ..	,,	,, ..	,, ..	,,	Fallow ..	Oats
1942 ..	Wheat ..	Wheat ..	Wheat ..	Wheat ..	Wheat ..	Wheat ..	Wheat ..	Wheat
Percentage of take-all ..	14·6	2·2	0	13·2	76·4	72·1	3·4	9·7
Grain yield per ear (grams) ..	5·39	1·96	7·63	2·65	1·04	0·84	4·22	3·52
Yield in bushels per acre ..	30	*Not harvested	38	*Not harvested	*Not harvested	*Not harvested	30	25
Mean height per plant (cm.) ..	46·8	35·6	49·2	37·8	21·0	21·7	46·4	43·3
Mean number of tillers per plant ..	3·07	1·44	4·08	2·15	2·02	2·1	2·83	2·31

* Not enough grain to warrant harvesting.

2. Results

The data for the effect of each of the treatments as revealed in the 1942 season are shown in Table 1.

It will be seen that take-all occurred in all treatments except where bare fallowing was practised for two years (plot 3). The severity of the disease was greatest in the plots continuously cropped to wheat (plots 5 and 6). The occurrence of diseased plants in the two one-year fallow, plots 1 and 7, was noticeably in patches, indicating localized areas of infection. The plants in all plots showing take-all symptoms had variable numbers of roots with lesions. The greatest amount of root destruction occurred in plots 5 and 6 continuously cropped to wheat. All plants examined from the two years' bare fallow, plot 3, showed complete freedom from root lesions.

The well established beneficial effects of fallowing on the vigour and yield of the crops is borne out in the data on the height and tillering, and on yield in plots 1, 3, and 7. Plots 1 and 7 were not kept in the bare-fallow condition and many weeds, including grasses susceptible to *O. graminis,* such as *Hordeum leporinum, Bromus hordeaceus* and *Lolium perenne,* grew on these two plots during fallow. The presence of weeds in these plots served as carrier plants for *O. graminis,* and at the same time reduced the nutritional effects from fallowing. This is reflected in the occurrence of take-all and lower yield in both plots. In plot 3, which was maintained in the bare-fallow condition for two years, the highest yield (38 bushels per acre) of all the treatments was obtained. This appears to be due partly to the eradication of take-all from the soil by starving out the fungus, and partly to the conservation of soil moisture and increase in available plant nutrients through fallowing.

In plots 2, 4, 5, and 6, the crop was so poor that there was not enough grain to harvest. In plot 2, where wheat was sown after two years under ryegrass and subterranean clover pasture, there was too much competition between the ryegrass and wheat from the time of germination until heading out. In plots 5 and 6, continuously cropped to wheat, the two-fold effect of depletion of soil nutrients and the high incidence of take-all was apparent. In addition there was competition from weeds in these two plots. In plot 4, which was under lucerne for two years, the drying of the subsoil and competition from weeds appeared to be the main factors contributing to the failure of the crop on this plot.

3. Discussion

These results provide further evidence that the presence of *Ophiobolus graminis* in the soil is directly related to the occurrence of take-all affected plants. At the same time, the effect of soil fertility on crop vigour in relation to the severity of the disease is apparent.

It is well established that practices inducing high soil fertility, such as fallowing, rotational cropping, and the addition of fertilizers, favour crown root development, which in plants attacked by *O. graminis* would ensure replacement of roots destroyed by fungal attack, and thereby provide the plant with a better chance of survival. No experimental evidence has yet been forthcoming to suggest that

the roots of wheat are immune from attack by *O. graminis*. Furthermore, the writer has frequently obtained evidence which fully substantiates the statement made by Garrett (1938) that "the fungus can attack the most vigorously growing plants, and there is no question of the plant having to be predisposed to the disease by unfavourable conditions."

Therefore, it is concluded that if all the roots of a plant were free from lesions, either *O. graminis* was not present or conditions were unfavourable for the growth of the fungus.

In the two years' bare-fallow treatment reported above, conditions would have been favourable for the growth of *O. graminis*, as shown by the activity of the fungus in the blocks on either side of this treatment. The roots of all the plants examined from this plot were free from lesions. It is assumed, therefore, that the fungus was not present in the soil, and that as a result of the long bare-fallowing treatment the fungus was eradicated. Of all the treatments, the yield and vigour was greatest in the two years' bare-fallow treatment. This result would appear to be due to enhanced soil fertility and conserved subsoil moisture.

Agronomic practices tending to reduce soil fertility such as continuously cropping to wheat, competition from weeds, and low soil moisture would create conditions more favourable to the development of take-all symptoms because these conditions do not favour root development, and attacked plants are unable to replace roots destroyed by the fungus. In the results reported above, soil fertility was lowered in the blocks continuously cropped to wheat for four years, in the treatment under pasture for two years, and in the treatment under lucerne for two years. However, competition for soil moisture was responsible for crop failure in the latter two treatments. In the pasture treatment annual ryegrass was used, and normally seed setting and germination of this crop would have been poor in Canberra because of the dry summer. The 1941–42 summer was a wet one, and resulted in the setting of ryegrass seed and good germination of seed in autumn after the plot had been cultivated in preparation for the wheat sowing of 1942. In the lucerne treatment the complete removal of lucerne crowns was not effected, and the weed population on this plot was high; these competed with the wheat.

In the plots continuously cropped to wheat, take-all was severe and crop failure was due in part to disease and to impoverished soil conditions and competition from weeds.

4. General Conclusions

In the light of the results reported in this series of studies on the etiology of the disease at Canberra, and those reported in the literature, the following conclusions on the incidence and severity of take-all disease of wheat may be made.

The incidence of the disease depends on the presence of the fungus *Ophiobolus graminis* in the soil, and, in turn, the presence of the fungus is related to the previous history of the field. Practices conducive to its persistence in the soil are the continued growing of susceptible crops such as wheat, barley, and certain pasture grasses, and favourable soil conditions such as light-textured soil (Garrett,

1942), and high soil nitrogen content (Garrett, 1944). Practices leading to the disappearance of the fungus from the soil are the growing of non-susceptible crops such as oats, bare-fallowing, and rendering the soil conditions unfavourable by compacting the seed bed (Garrett, 1942), and by increasing biological antagonism through increasing the carbon content of the soil (D'Aeth, 1939).

The severity of the disease is influenced primarily by the capacity of the attacked wheat plant to outstrip root destruction with new root development, and by the distribution of inoculum in the soil. On the one hand, root destruction is influenced by the virulence of the strain of *O. graminis* (White and McIntyre, 1943), which was shown to be genetical in origin (White, 1942), and by the soil conditions affecting the rate of growth and spread of the fungus (Garrett, 1937; Winter, 1939). On the other hand, root formation is influenced by soil fertility, soil moisture, and the surviving crown root primordia. The effectiveness of the balance between destruction of roots and root formation in maintaining the water supply and nutrition to the affected plants depends on the relative rates of root destruction and formation, soil moisture, and the demands of the shoot system on the functioning root system (White, 1947).

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The Treatment of Cut Potato Setts with Zinc Oxide

1. Condition of the setts, growth, and yield

By J. G. Bald, M.Agr.Sc., Ph.D.*

Summary.

In this and a succeeding paper, an analysis is made of data from a field trial on the effects of treating cut potato setts with zinc oxide before planting. Treatments with water and an organic mercury dip served as controls.

Properly used, the zinc oxide reduced premature rotting of the cut potato setts. It rather encouraged than hindered suberization. Parallel mercury treatments damaged the cut surfaces and allowed rotting organisms to gain entrance to the tuber tissues. In no treatment was there more than 6 per cent. of misses.

The most efficient zinc oxide treatment had no effect on emergence; others slightly retarded emergence. Mercury treatments applied to the cut surfaces seriously retarded emergence.

Zinc oxide treatments had relatively little effect on the leaf area of the plants; mercury treatments caused serious reductions in leaf area. Possibly the slight effects of zinc oxide treatments were partly due to absorption of zinc through the sett.

Zinc oxide treatments had slight effects on the total yield; mercury treatments depressed the yield. Careful analysis suggested that the zinc absorbed through the sett increased the ratio of tubers to foliage, possibly because the soil in which the plants were grown was slightly deficient in zinc. Treatments that encouraged premature rotting of the setts depressed the total yield. Zinc oxide dip efficiently applied increased the over-all growth of the plant by protecting the setts from premature rotting.

1. Introduction

Zinc oxide was found in laboratory trials to act as a protector of the cut surfaces of potato tubers against rotting organisms (Bald, 1943a). In contrast to copper compounds, it rather aided than retarded suberization. Dillon-Weston and Taylor (1944) found that salts of manganese, in addition to zinc salts, could be applied to the cut surfaces without adversely affecting suberization. In a South Australian field trial of zinc oxide as a means of controlling missing in potato crops (Beare, 1944), it was incidentally found to give some control of common scab caused by *Actinomyces scabies*. The trial gave no information about missing, because there were few misses amongst the untreated controls.

As a means of protecting cut surfaces of potato tubers in greenhouse and laboratory work, dipping in zinc oxide suspensions and suberization on damp sand have been very successful. In a recent development of experimental methods involving the removal of single eyes with the minimum of tissue from dormant tubers, zinc oxide has again given effective protection of the cut surfaces. The single eyes are dipped in zinc oxide suspension and placed in petri dishes on

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damp filter paper impregnated with zinc oxide or in trays of damp sand. Even in the saturated atmosphere of the closed petri dish they generally remain clean and turgid, and sprout very rapidly (Fig. 1)*.
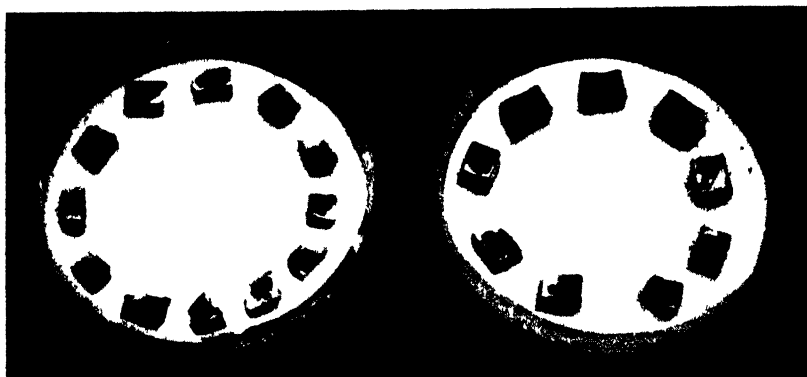
Fig. 1.—Single eyes from potato tubers, treated with zinc oxide and sprouting in petri dishes.

2. Description of the Trial

In the dry spring of 1945, conditions apparently favourable for missing in the potato crops had developed at Canberra. A field trial planted with cut seed pieces was planned to test the effect of zinc oxide on missing, on *Rhizoctonia* infection, with which missing may sometimes be associated, and possibly on common scab. Tubers of the variety Up-to-Date, carrying medium to heavy infection with *Rhizoctonia* and scab lesions, were sorted out from reject material of the previous crop. Tubers as clean and sound as were available, but, unfortunately, not from the same original source, were chosen for an untreated control.

Various treatments with zinc oxide, and an organic mercury compound, were applied to the infected tubers, and the trial was planted on fallowed ground at the C.S.I.R. Experiment Station at Dickson, during the third week of October. The fallowed soil was moister than had been expected after so long a dry period, and within a week of planting 1·4 inches of rain fell. The soil conditions, instead of inducing a high percentage of misses, gave a high percentage of emergence, even in instances where treatment had injured the setts.

The effects of *Rhizoctonia* became evident in the foliage soon after flowering, but a confusion of symptoms was produced by infestation with Rutherglen bug (*Nysius vinitor*) and a severe attack of spotted wilt, both seasonal and unexpected. These prevented records of the incidence of *Rhizoctonia* from foliage symptoms. Fortunately, as the bug infestation and the spotted wilt were distributed over all treatments, they had little effect on the estimation of tuber infection, or on the estimation of infection with common scab. The latter was present on a large proportion of the tubers, and data were obtained for the effects of treatment on both diseases. These are reported in a succeeding paper.

* Mr. D. O. Norris has found sprouting is encouraged by keeping the petri dishes in the dark at a temperature of 25° C.

The sequence of operations and the observations made during the trial were as follows:

October 18.—The treatment of tubers was begun.

October 23, 24.—The trial was planted.

November 9.—The first plants began to emerge.

November 22.—Records were taken of the numbers of plants emerged.

November 28 and 30.—The leaf area of every plant above-ground was recorded (Bald, 1943b).

November 29.—Two plants from each of 104 plots were dug, setts were rated for the condition of the cut surface and the extent of rotting; the extent of the lesions on each stem and on the stolons were also noted. The lesions were of the type usually associated with *Rhizoctonia*. Records were also taken of the presence of macroscopically visible runner hyphae on the underground stems and stolons.

January 21.—The leaf area of each plant was recorded as the haulms approached their maximum size.

March 28.—A few plants from all treatments were dug, and the tubers examined for the extent of infection with *Rhizoctonia* and scab, and the type of data that might be obtained from the yields.

April 5–16.—The trial was dug. The haulms were dead, as the growing season had been slightly shortened by an attack of early blight. Thereafter the tubers were sorted, weighed, and examined.

3. Treatments

In all, there were thirteen treatments in the trial. They were:—

1. Clean tubers, cut October 22, setts dipped in water, put in small cloth bags, sufficient in each to plant one plot of the trial. The cloth bags were covered with damp sacks to maintain humidity until the setts were planted during the following two days, October 23 and 24.

2. Infected tubers, cut October 18, setts dipped in water, tipped into open trays and allowed to dry out. The setts lay two to three deep, the cut surfaces frequently in contact with the skin of other setts. On October 22 they were put into small cloth bags, which were covered with damp sacks. A proportion of setts were definitely shrunken after the four days drying.

3. Infected tubers, dipped whole in water on October 18, left in trays until October 22, cut, bagged, protected by damp sacks.

4. Infected tubers, cut October 22, dipped in water, bagged, protected by damp sacks.

5. As for 2, but on October 18 dipped in zinc oxide suspension.

6. As for 3, but on October 18 dipped in zinc oxide suspension.

7. As for 4, but on October 22 dipped in zinc oxide suspension.

8, 9, 10. As for 2, 3, 4, but dipped in an organic mercury fungicide, improved Hortosan.

11, 12, 13. As for 2, 3, 4, but dusted with a zinc oxide-DDT dust.

The zinc oxide suspension consisted of 5 oz. zinc oxide in 1 gallon of water; the organic mercury dip was made up according to directions; and the dust contained 2 per cent. zinc oxide and 1 per cent. DDT in

' pyrophyllite " filler. It was added to the setts in a barrel duster at the rate of 1 oz. to 8 lb. of setts, and the duster was turned until coverage of the setts was complete.

Treatments with the dust were included because zinc oxide had been added to a DDT pyrophyllite dust used by Mr. G. A. Helson in trials on methods for controlling potato moth (*Phthorimaea operculella* Zell.). The zinc oxide largely prevented the rots that normally follow damage by grubs of the potato moth.

The trial consisted of two symmetrical incomplete blocks planted side by side with the rows continuous. There were eight replications of each treatment. Each treatment was a single row 1 chain in length, containing 32 plants equally spaced. At harvest this number had been reduced to a row length of 28, because plants had previously been taken from a strip of 4 across each block. The rows were slightly more than 3 feet apart.

Fertilizer, 6 : 1 superphosphate and ammonium sulphate, was applied in the furrow at the rate of 2 cwt. per acre. The plot was furrow-irrigated twice during the season, on December 17 and January 7. The aggregate amount of water delivered was approximately 4 inches. The rainfall was more regular than is usual during that period of the year; the aggregate between digging and planting was 12·4 inches. There were the usual hot summer temperatures and drying winds.

4. Examination of Setts

(i) *Description of Material and Rating Methods.*

On November 29 and 30, five weeks after planting, two plants in each of the 104 plots of the trial were dug, and the setts, underground stems, and stolons were examined. The setts were present on all plants; some were sound and some rotting.

The main shoots of the plants were still extending; at the top of some of them young buds could be seen in the terminal rosette of leaves. Stolons were growing out, and the underground stems were sappy and firm.

The condition of the sett on each sampled plant was classified according to the following categories:

1. Cut surface firm, clean, and well suberized.

2. Cut surface firm, but suberization relatively poor, giving evidence of fungal growth. Sometimes the surface was covered with a corky, powdery layer of dead tissue permeated by fungus hyphae.

3. Cut surface pitted, often at the edge, and rot beginning.

4. Rot well established.

It was found that the first class might have been subdivided according to the solidity and freshness of the suberized surface. The condition of most setts treated with zinc oxide the day before planting (treatment 7) was distinctly better than any of the others. This does not appear from the rating values. The second rating class was also heterogeneous. It included setts on which extensive chemical injury to the cut surface had killed the tissues to a depth of 5 mm., and tubers of which the suberized layer was merely discoloured and easily rubbed off. There were various intermediate stages of injury and inefficient suberization.

A value of 1, 2, 3, or 4 was given to each sett according to the category into which it fell. Values for the two setts dug from each plot were added, and with this sum as a plot total a full analysis of differences in the condition of the setts was made. The treatment means, halved to give average ratings for a single sett, are in Table 1. Beside them are the actual numbers of setts in each of the four rating classes. Also in this table are those portions of the rating totals represented by tubers in which rots were actually present, i.e. tubers that fell into classes 3 and 4.

A mean rating of 1 for all setts sampled from a treatment would imply that every one of the 16 setts was perfectly sound, with no obvious sign of fungal growth on the cut surface. A mean of 4 would imply that every one was rotting and the rot was well advanced.

TABLE 1.—CONDITION OF THE SETTS SAMPLED FIVE WEEKS
AFTER PLANTING.

Treatment		Number of Setts Rated—				Mean Rating, Single Sett	Sum of Ratings Rot Only
Description	Number	1	2.	3	4		
Clean setts	1	13	3	0	0	1 20	0
Infected setts, no treatment	2	8	2	2	4	2 15	22
	3	7	3	3	3	2 08	21
	4	7	8	1	0	1 69	3
Infected setts, zinc oxide dip	5	7	1	5	3	2 19	27
	6	8	5	3	0	1 70	9
	7	16	0	0	0	1 04	0
Infected setts, mercury dip	8*	0 (0, 0)	8 (1, 7)	4 (4, 0)	4 (3, 1)	2 78	28
	9	11	4	1	0	1 34	3
	10	2	13	1	0	1 92	3
Infected setts, zinc oxide DDT dust	11	6	1	3	6	2 54	33
	12	9	3	2	2	1 83	14
	13	9	3	1	3	1 85	15
Significant difference			.			0 77	10 6†

* Duplicate ratings for treatment 8 in the two main blocks of the trial.
† Difference does not apply to mercury treatments, see text.

(ii) *Controls.*

Treatment 1, clean setts treated only with water (called hereafter untreated setts) had no evidence of rot, and very little fungal growth on the cut surfaces of the setts. By contrast, the controls carrying *Rhizoctonia* sclerotia and scab lesions (2, 3, 4) had higher ratings, i.e. significant numbers of setts with evidence of fungal action on the surfaces, or actual rotting. Although there was not the full range of methods of application among the controls planted with clean setts, this contrast suggests that much of the infection may have been tuber-borne and did not arise from the soil.

Throughout the following discussion it is assumed that a large proportion of the organisms causing rotting of the setts was derived from tuber-borne infection. The rotting invariably began from the cut surfaces. Apparently the exposed tissues provided suitable and convenient sites for the growth of organisms originally carried externally on the skin of the tubers. No assumption is made about the identity of these organisms, but it seems likely that strains of *Rhizoctonia solani* were among those causing damage to the setts.

The comparisons between treatment 4 and treatments 2 and 3 reveal interesting differences in the condition of the setts. The mean rating of 1·69 for setts in treatment 4 was mainly due to a rating of 2 for 8 of the 16 setts. On these 8 setts suberization had been obstructed by organisms that had invaded the cut surface, but up to the time of sampling the suberized cells had been an effective barrier against actual rotting. Suberization had been just sufficiently rapid to protect the underlying tissues. On only one sett had it been ineffective.

In treatments 2 and 3 approximately the same number of setts gave evidence of obstruction to suberization by invading organisms as in treatment 4, but in each of treatments 2 and 3, 6 setts were definitely rotting, as against 1 in treatment 4. Although the mean ratings for setts in the three treatments were not significantly different, the differences between those portions of the rating totals contributed by setts that were actually rotting were clearly significant (see last column of Table 1). The differences between these ratings for treatments 4 and treatments 2 and 3 were 19 and 18, and the minimum significant difference was 10·6.

The operation that appeared to give this advantage to the setts in treatment 4 was the wetting of the cut surfaces and the maintenance of high humidity around the setts until they were planted. The setts in treatment 2 were also dipped in water, but were allowed to dry out for 4 days before planting. Those in treatment 3 were cut the day before planting, and kept under moderately humid conditions, but were not wetted.

(iii) *Zinc Oxide Dip.*

Of the treatments in which the setts or tubers were dipped in a suspension of zinc oxide, number 7, dipping of the cut setts the day before planting, completely prevented premature rotting. It gave the cut surfaces an extra firmness and solidity, and there was practically no evidence of obstruction to suberization from fungal activity. This treatment had no discernible effect on emergence, although the zinc oxide dipping and dusting treatments as a whole caused a slight delay in emergence. The growth of the plants in treatment 7 was as good as in any other treatment (Tables 3A, 3B).

. When the zinc oxide dip was applied to the setts after cutting, and they were allowed to dry out for 4 days, it gave no protection against rotting (compare treatments 5 and 2). Applied only to the whole tubers, it gave the setts cut from them some protection (treatment 6). Although the mean rating for treatment 6 was not significantly less than for the corresponding control (treatment 3), the sum of ratings for only those setts in which rot was present was

significantly less (last column, Table 1). As in treatment 4, suberization in most instances more than kept pace with fungal invasion, even though on 5 of the 16 setts there was evidence of some fungal growth and destruction of surface cells (five setts were rated 2).

(iv) *Organic Mercury Dip.*

The mercury treatments produced a different picture. The setts of treatment 8 were badly shrunken and discoloured when they were planted; and, when the sampled plants were dug 5 weeks later, every one showed evidence of chemical injury. The cut surfaces carried a superficial layer, sometimes 5 or 6 mm. thick, of brown powdery dead tissue permeated by the mycelia of saprophytic fungi. The surface of the solid tissue below was brown, and carried no properly suberized layer. Possibly the main barrier against rotting organisms on those setts was the antagonistic action of the saprophytic organisms in the dead tissue; certainly normal barriers were almost absent.

This resulted in a condition of instability which was reflected in the duplicate rating totals for the two main blocks of the trial (Table 1, treatment 8, figures in brackets). Although only distances of 4 to 12 feet separated sampled plants in the two blocks, the line of division between the two blocks was also, along much of its length, the approximate boundary between two soil types. The soil of one main block was a clay loam, of the other a clay loam mixed with a high proportion of gravel. Of the eight setts of treatment 8 dug from the former soil, seven were rotting. Of the eight setts dug from the gravelly soil, only one was rotting. There is little doubt this variability within the same treatment represented a real difference between blocks in the reaction of the setts. Changes in soil conditions that did not affect the incidence of rot in setts from other treatments affected it in this one.

An incidental result of this instability was that the values for the mercury treatments were discarded from the estimate of the standard error for the rating totals, rotted tubers only. Hence the significant difference, 10·6, is not applicable to comparisons involving the mercury treatments. It is probable, however, that the condition of setts in treatment 9 was better than in the corresponding control (3). The difference between the mean ratings for 3 and 9 was almost at the 1 in 20 level of significance. There can be no doubt that the poor condition of the setts in treatment 10 was due largely to chemical injury.

Chemical injury to the setts in treatments 8 and 10 was followed by delayed emergence and a reduction in size of the plants, particularly in treatment 10. The high humidity maintained after the setts in treatment 10 were dipped apparently enabled the absorption of greater quantities of mercury at the cut surface.

The mercury dip, applied to whole tubers, which were then allowed to dry out before cutting (treatment 9) improved the condition of the sett in comparison with the corresponding control, treatment 3, but it cannot be considered so efficient for this purpose as treatment 7. Although the difference between treatments 7 and 9, as shown in Table 1, is not significant, the qualitative differences in the condition of the setts, that found no expression through the rating scale, were in favour of this zinc oxide treatment.

(v) *Zinc Oxide-DDT Dust.*

The application of the zinc oxide-DDT dust did nothing to improve the condition of the setts. The ratings for these treatments (11, 12, 13) were no less than those for the corresponding controls (2, 3, 4), and the ratings for rotted setts only were significantly higher than those for the corresponding zinc oxide treatments (5, 6, 7). Only the treatment in which the dust was not put directly on the cut surface remained free from this effect. In that one (12) the sum of the ratings for rotted tubers only was not significantly different from either the corresponding zinc oxide dip treatment (6) or the corresponding control (3).

The dust probably acted by withdrawing water from the cut surfaces of the setts and hindering rapid suberization, without preventing the entry of rot organisms. That dusted setts rapidly lost water was evident from the condition of the setts in treatment 11, four days after cutting and dusting. They were as badly shrunken as the setts of the corresponding mercury treatment (8), although the surfaces were not as seriously discoloured.

(vi) *Summary.*

Rapid and efficient suberization appeared, under the conditions of this trial, to be essential for the prevention of premature rotting. A sufficient and continuous water supply to the exposed cells of the cut surfaces was absolutely necessary for efficient suberization. Wetting the cut surfaces and the maintenance of humid conditions until planting in damp soil gave these cells the water they needed. Also it may have encouraged the growth of organisms which found their way from the skin of infected setts to the cut surfaces of others in contact with them, or grew over the edge of the cut surface from the skin of the same sett. In spite of this invasion, rapid and efficient suberization in most instances gave the underlying tissues of treated setts sufficient protection from fungal attack.

The role of the zinc oxide appeared to be fungistatic rather than fungicidal. Under humid conditions it discouraged the growth of rotting organisms sufficiently to allow full suberization. Under conditions of insufficient water supply to the cells of the cut surface, zinc oxide gave little or no protection. It may possibly have retarded suberization by encouraging the loss of water from the exposed cells, but in tne presence of sufficient water it rather encouraged than discouraged suberization. There was no evidence that it had any toxic action on the potato tissue.

Under similar conditions the organic mercury dip, improved Hortosan, was toxic to the cells of the potato tuber, and probably also to the rotting organisms. For this reason, applied to whole tubers, it gave some protection to the setts cut from them, without noticeably affecting suberization, but its effect on the cut surfaces was to destroy many layers of cells, and its absorption seriously affected the emergence and subsequent growth of the potato shoots.

5. Emergence

The emergence data are in Table 2. These are the actual numbers of plants above ground on three dates, November 22, 13 days after the first shoots were observed breaking through the soil, and on November 28 and January 23, when leaf area ratings were made. On November 22, 247 plants had emerged from the 256 setts planted in treatment 1 (97 per cent.). Emergence for the corresponding control with infected seed (treatment 4) was similar. The zinc oxide treatments (5, 6, 7) and (11, 12, 13) on the whole were slightly behind the untreated controls (1 and 2, 3, 4). The treatments in which the cut setts were treated with the mercury dip were considerably behind (8, 10). The most severely affected was treatment 10, in which the cut setts were dipped the day before planting began, and remained under humid conditions until they were planted. Allowing the setts to dry out after dipping (8) caused less retardation of emergence, but finally about the same proportion of misses (6 per cent.).

Apart from the mercury dip series, timing of the treatments had slight but uniform effects. When the tubers were dipped or dusted the day before planting and kept under moist sacks, they sprouted most promptly. The slowest to emerge were those dipped or dusted whole, left for 4 days, and cut the day before planting. Those cut and treated several days before planting were intermediate.

TABLE 2.—NUMBERS OF PLANTS EMERGED ON THREE DATES, FIVE WEEKS AFTER PLANTING; WHEN THE FIRST LEAF AREA RATING WAS MADE; AND AT MATURITY WHEN THE SECOND LEAF AREA RATING WAS MADE.

Treatment.	Number of Plants		
	November 22	November 28	January 23
1	247	252	256
2	238	249	251
3	234	248	253
4	243	255	256
5	229	245	247
6	226	248	251
7	237	246	252
8	179	229	239
9	214	245	251
10	93	184	240
11	227	248	249
12	219	241	247
13	233	246	248

6. Leaf Area

The results of rating for leaf area are in Table 3. Each plant was rated according to an established scale (Bald, 1943b). The ratings within each plot were added without conversion to actual areas, and the total plot ratings were used as the unit for analysis. No direct allowance was made for missing plants.

The plot totals were very nearly proportional to the logarithms of the total leaf area of each plot. Comparing increments of leaf area ratings over a period of time was more nearly equivalent to a

comparison of growth rates than of increments in leaf area. It was for this reason, and to eliminate the labour of converting the ratings to areas, that summed ratings were used for analysis.

The means for treatments in Table 3 show significant differences. The plants in treatment 1, following rapid emergence, gave the highest values for leaf area on November 28, but not on January 31. This is a reflection of the difference in the source of seed tubers used to obtain clean setts for treatment 1 (p. 88): the difference is evident throughout the growth and yield data. It affected all comparisons with treatment 1 involving growth and yield, but not those that were confined to the incidence of disease. It arose from the early maturity of the only Up-to-Date clone of which clean tubers were available when the zinc oxide trial was planned. The discard Up-to-Date tubers used for treatments 2–13 were unlabelled, and their exact origin was unknown, but they were found from the results of the trial itself, to be derived from later maturing clones. Care was taken to mix the tubers assigned to these 12 treatments very thoroughly before they were divided into single lots.

TABLE 3A.—LEAF AREA, NOVEMBER 28. MEAN TOTAL RATINGS FOR SINGLE PLOTS.

Tr.	Leaf Area.	Tr.	Leaf Area	Tr.	Leaf Area.	Tr.	Leaf Area.	Tr.	Leaf Area.	Mean Leaf Area.
..	..	2	188·4	5	186·5	8	129·9	11	169·1	168·5
..	..	3	204·1	6	196·0	9	184·2	12	182·7	191·8
1	227·7	4	209·4	7	210·8	10	83·8	13	193·3	174·3

Mean leaf area 200·6   197·8   132·6   181·7

Significant difference between two treatment means   ..   .. 16·0

TABLE 3B.—LEAF AREA, JANUARY 23. MEAN TOTAL RATINGS FOR SINGLE PLOTS.

Tr.	Leaf Area.	Tr.	Leaf Area.	Tr.	Leaf Area.	Tr.	Leaf Area.	Tr.	Leaf Area.	Mean Leaf Area.
..	..	2	489·5	5	474·9	8	397·8	11	458·8	455·2
..	..	3	502·1	6	484·3	9	470·5	12	471·1	482·0
1	493·2	4	526·0	7	510·1	10	359·7	13	493·9	472·4

Mean leaf area 505·9   489·7   409·3   474·6

Significant difference between two treatment means   ..   .. 28·7

In spite of the slight lag in emergence, the plants in the zinc oxide dip series (5, 6, 7) were not significantly smaller either on November 28 or January 23 than the controls (2, 3, 4), although on January 23 the difference was close to significance. The zinc-oxide-DDT dust treatments (11, 12, 13) caused a slight but definite reduction in leaf area. The mercury treatment, 9, in which tubers were treated whole and left 4 days before cutting, gave almost exactly the same leaf area values as the corresponding zinc-oxide-DDT dust treatment (12). The other two mercury treatments 8 and 10, particularly 10, had by far the lowest leaf areas on November 28. The leaf areas reflected the delay in emergence caused by treatment of the cut surfaces. The difference was reduced, but by no means eliminated, on January 23.

There were other effects on leaf area than those resulting from delayed germination. To disentangle them from the available data was difficult. The effects of delayed germination were not easily defined or computed, as they were probably both direct and indirect. Firstly the growth cycle as a whole was shifted in time, and this may have had secondary effects on the ultimate size of the plants. There were small differences, up to 6 per cent., in the numbers of plants finally emerging, and there were the unpredictable results of Rutherglen bug invasion, infection with spotted wilt, etc. The bugs, for example, appeared to attack and injure young plants more severely than old, causing more damage to late-emerging plots, and probably inducing differences between treatments in growth rate.

The most practicable objective method of obtaining an estimate of treatment differences, freed from the effects of differential germination, was the use of regressions for leaf area on the emergence count of November 22. The results can be used only as a guide to what may have happened, not as convincing evidence of what did happen. It is possible that under- or over-correction of particular treatment means might produce differences between the estimated values that did not exist in fact.

The relation between leaf area and emergence was close, but not linear. Regressions of the form

$$Y = a + bx + cx^2$$

were calculated for, (1) leaf area, November 28, on emergence counts, November 21, and (2) leaf area, January 23, on emergence counts, November 21. The regression equations were used to estimate what the leaf areas would have been if all treatments had germinated at the same time. The variance of the estimated values was less than that of the unmodified values; but no figure for significant differences is given, for fear of surrounding the results with a spurious air of certainty. The estimated treatment means are in Table 4, presented in the form of deviations from the calculated regression curve. Negative values were below, positive values above the curve.

In this table are also the mean values for the four complete series of treatments, and the means for the three different methods of application or timing. Tratment 1 was omitted from the calculation of the means. Means of several treatments are less likely than means for single treatments to have been distorted by under- or over-correction, so discussion will be confined to them.

TABLE 4A.—LEAF AREA, NOVEMBER 28, ESTIMATED FOR CONSTANT EMERGENCE. VALUES GIVEN AS DEVIATIONS ABOVE OR BELOW THE REGRESSION CURVE FOR LEAF AREA AND NUMBER OF PLANTS EMERGED BY NOVEMBER 21.

Tr.	Leaf Area.	Tr.	Leaf Area	Tr.	Leaf Area.	Tr.	Leaf Area	Tr.	Leaf Area.	Mean Leaf Area.
..	..	2	−55·0	5	−17·6	8	−15·9	11	−74·6	−40·8
..	..	3	36·3	6	36·0	9	36·9	12	16·8	31·5
1	29·0	4	−8·9	7	22·5	10	3·3	13	−8·7	2·1
	Mean leaf area	−9 2			13 6		8·1		−22·2	

TABLE 4B.—LEAF AREA, JANUARY 23, ESTIMATED FOR CONSTANT EMERGENCE. VALUES AS ABOVE.

Tr.	Leaf Area.	Tr	Leaf Area	Tr	Leaf Area	Tr.	Leaf Area	Tr.	Leaf Area.	Mean Leaf Area.
..	..	2	−40·8	5	−41·1	8	−59·4	11	−88·5	−57·5
..	..	3	96·7	6	18·1	9	23·8	12	8·3	36·7
1	−121·7	4	55·9	7	125·3	10	12·3	13	11·6	51·3
	Mean leaf area	37·3			34·1		−7 8		−22·9	

Methods of application had considerable effects on the growth of the haulm. Throughout the period of growth represented by the two leaf area ratings, the treatments in which the tubers were cut, and the setts then treated and allowed to dry out for 4 days (2, 5, 8, 11), resulted in smaller haulms than those in which the setts were cut and treated just before planting (4, 7, 10, 13). The other method of application, in which the whole tubers were treated before the setts were cut (3, 6, 9, 12), produced at an early stage larger haulms (Table 4A), and at a later stage smaller haulms (Table 4B) than treatments 4, 7, 10, 13. There was here a suggestion that conditions induced in a sett by treatment affected the growth rate of the plants as well as the germination, possibly because they affected the ratio between the tops and tubers. The means for groups of three treatments suggested smaller but similar variations in size and growth rate. This matter will be discussed again in a later section. In the meantime, it is clear that more vigorous growth as well as better germination was obtained from treatments which left the setts fully turgid and well suberized at planting.

7. Total Yield

The yield of the trial as a whole was fairly high, about 9 tons per acre, but the tubers produced were far below commercial standard. The incidence of spotted wilt, the check to growth induced by Rutherglen bug, scab, *Rhizoctonia,* and some injury from the potato moth all reduced the proportion of the better grade tubers. The defects in quality, however, did not affect the comparisons between total yields for different treatments.

TABLE 5.—TOTAL YIELD. TREATMENT MEANS IN LB. PER PLOT.

Tr.	Yield	Tr.	Yield.	Tr	Yield.	Tr.	Yield	Tr	Yield	Mean Yield.
..	..	2	77·4	5	79·1	8	56·9	11	70·4	71·0
.:	..	3	81·6	6	82·3	9	73·7	12	76·1	78·4
1	77·2	4	85·4	7	87·0	10	52·3	13	81 6	76·6
	Mean leaf area	81·5		82·8		61·0		76·0		

Significant difference between two treatment means

   These are summarized in Table 5. Because of the different origin of the seed tubers used for the setts of treatment 1, the yield was below that of the corresponding control planted with infected setts (4). The zinc oxide dip treatments (5, 6, 7) taken together gave a mean yield very similar to that for the controls (2, 3, 4); the zinc oxide-DDT dust caused a slight reduction in total yield (11, 12, 13), and the mercury treatments caused a greater reduction (8, 9, 10). Of the three mercury treatments, those in which the cut setts were treated (8, 10) yielded 28 and 40 per cent. less than the corresponding controls. The reduction due to treating whole tubers with the mercury dip before cutting (9) was significant, but only 10 per cent.

   The reductions in yield could in a large measure be traced back to retarded germination, but probably also the nature of the treatment and the condition of the setts had an influence on yield independent of germination. In the values for total yield and leaf area at maturity (Tables 5 and 4B) there are indications that plants from setts treated with zinc oxide yielded somewhat better than plants of similar mean size amongst the control and mercury treatments. In Fig. 2, yield is plotted against leaf areas on January 23. A freehand curve is drawn through the values for mercury treatments and controls. All values for zinc treatments are above this line, and the mean difference between the six zinc treatments and points on the curve representing the same values for leaf area appears to be well beyond the 1 in 20 level of significance.

   There was apparently not the same balance between the production of foliage and tubers in the zinc treatments and the controls. Amongst plants with equivalent leaf area on January 23, those treated with

zinc oxide produced greater yields than the infected controls, but on the whole their leaf area was probably less. The mean reduction in leaf area below the controls (2, 3, 4) of the zinc oxide dip series (5, 6, 7) was 16·1, and the difference necessary for significance was 16·5. Both yield and leaf area of the zinc oxide-DDT series were less. The basic cause of these variations is not clear from the figures. The difficulties of disentangling the effects of interacting variables has already been mentioned in the section on leaf area. Similar methods as were described there may be used, with the same reservations, to analyse the parallel differences in leaf area and yield, and the possible reasons for them.
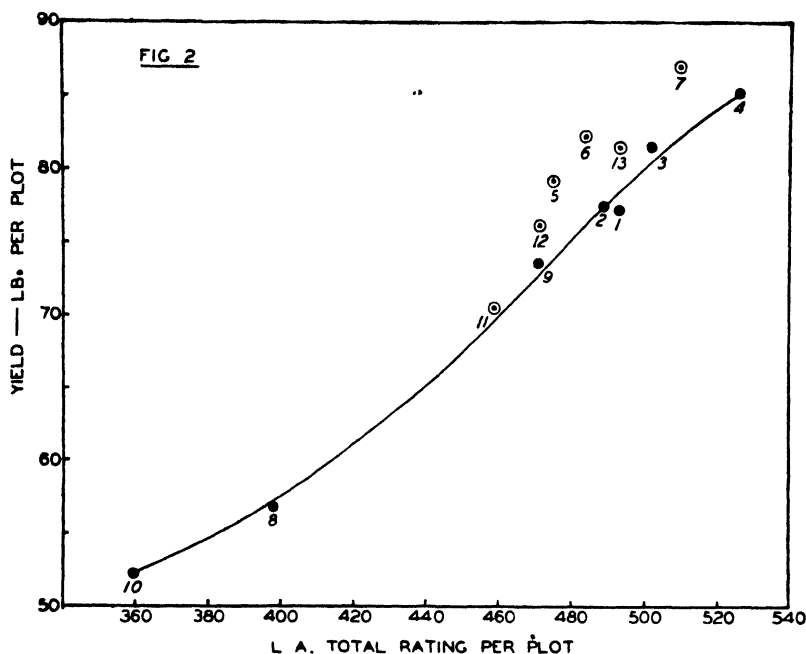

FIG. 2.—Mean total yield for 13 treatments plotted against maximum leaf area ratings. A freehand curve is drawn through the points for untreated controls (1, 2, 3, 4) and mercury treatments (8, 9, 10). Points for zinc oxide dip treatments (5, 6, and 7) and zinc oxide-DDT treatments (11, 12, and 13) are above the curve.

First of all, partial correlation coefficients were calculated between the leaf area ratings, November 28, and leaf area ratings, January 23. Both emergence and the square of emergence were eliminated from the coefficients because of the non-linear relations between emergence and leaf area. The partial correlation coefficient (0·5993) was s'f,-nificant, but much below the simple correlation, 0·9403. Independent of the effects of unequal emergence, there was some tendency for treatments in which the plants were small on November 28 still to contain small plants on January 23. The relative weakness of this underlying association suggests variations between treatments in growth rate during the intervening period. Even apart from the results of unequal emergence, the effects of treatment on the growth and development of the haulms were probably complex.

The association between leaf area on January 23 and yield was not so strongly affected by differences in emergence as that between leaf areas on November 28 and January 23. There was a very strong association, for treatment means within blocks, between leaf area on January 23 and yield ($r = 0.9667$, $n = 24$). The association was reduced, but not so greatly reduced as that between the two leaf area ratings, by eliminating the effects of unequal emergence ($r = 0.8611$). There was also a high degree of association between yields and the earlier leaf area ratings (November 28): how far this was dependent on unequal emergence was not tested.

These results may be interpreted as follows: Apart from the effects of unequal emergence the main effect of treatment was on the size of the plant as a whole. In addition, the ratio between haulms and tubers was probably affected. There was a close association between leaf area and yield, but not so close as to eliminate a reasonable chance that treatments had an effect on the ratio between the two.

8. Direct and Indirect Effects of Treatment

The possible effects of treatment outlined above were pursued by an extension of the analytical methods already adopted, and the results were put in graphical form. The effects of differential germination, the unwanted variable, were eliminated as far as possible from the yields as well as from both leaf areas by a regression equation of the second degree. The original criticism of this method still applies, that the estimated values for leaf area and yield may not have been consistently and adequately corrected for differential germination.

In Table 6 are the estimated mean yields for the 13 treatments, presented as deviations from the curves of the appropriate regression equations. The estimated values for leaf areas on November 28 and January 23 are in Tables 4A and 4B.

TABLE 6.—TOTAL YIELD, ESTIMATED FOR CONSTANT EMERGENCE. VALUES GIVEN AS DEVIATIONS ABOVE OR BELOW THE REGRESSION CURVE FOR YIELD ON NUMBER OF PLANTS EMERGED BY NOVEMBER 21.

Tr	Yield	Tr	Yield	Tr	Yield	Tr	Yield	Tr	Yield	Mean Yield
..	..	2	-13.2	5	5.9	8	-22.4	11	-25.6	-13.8
..	..	3	11.5	6	23.1	9	1.4	12	7.2	10.8
1	-33.9	4	8.7	7	22.2	10	4.3	13	10.8	11.5
	Mean yield ..	2.3		17.1		5.6		-2.5			

To obtain an accurate estimate of the relation between leaf area and yield, it would be necessary to integrate the leaf area over the period when tuber formation occurred. A crude approximation to such integration, useful for comparison between treatments, is the mean of the estimates for the two leaf area ratings. One of these was made at the inception of tuber formation, the other when the

plants were at or near their maximum size. Estimated yields were plotted against the mean of estimated leaf area ratings (Fig. 3), and below them were plotted the ratings for the condition of the setts (Table ⊥).

In Fig. 2, values for yield were plotted against leaf area on January 23 without eliminating the effects of unequal emergence, and the yields of the zinc oxide treatments were higher for equivalent leaf areas measured at that time. In Fig. 3 such differences appear more clearly defined. All treatments are within a relatively small range of estimated leaf area values, and the yields for the three methods of application within each main treatment fall about a curve that appears to be placed on a level characteristic for that treatment. The most interesting, and probably the most valid comparison is between the zinc oxide dip series (5, 6, 7) and the untreated controls (2, 3, 4). Points for the former occur uniformly above the latter, and correspondence in form between the curves drawn through the two sets of points is very close. Similar curves are drawn through the less regular series for the mercury dip and the zinc oxide-DDT series.
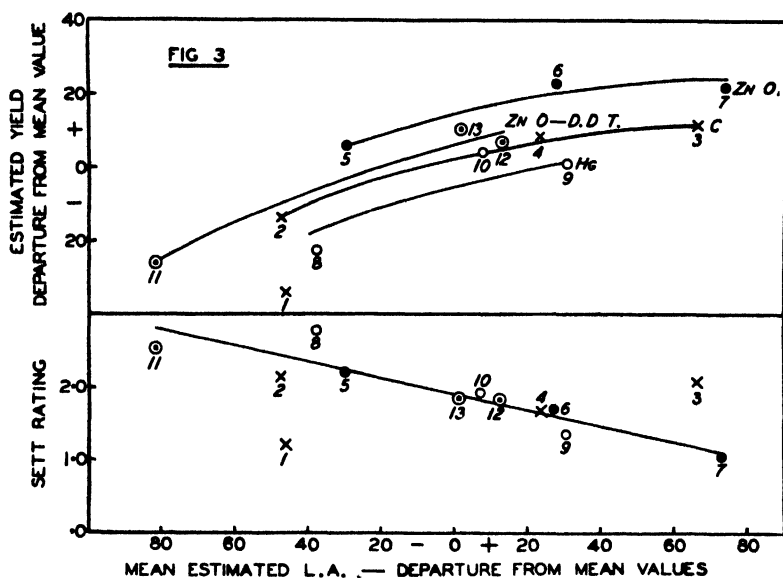
FIG. 3.

Of plants with the same mean leaf area during the period when tubers were forming, those growing from setts treated with the zinc oxide dip (5, 6, 7) produced a higher yield of tubers than those of the controls (2, 3, 4). Presumably zinc was absorbed by the treated plants in sufficient quantity to alter their metabolism. Those treated with zinc oxide-DDT dust (11, 12, 13) may have absorbed lesser amounts of zinc, which had effects so slight as to be of doubtful significance.

The lowering of yield for equivalent leaf area in the mercury treated series might be an artefact due to the degree of correction needed to bring the values to equal emergence. Against this view is

the position of the point for treatment 9; the correction needed to equalize for emergence in this instance was not great. If the difference between the mercury and control series was real, it would imply that the presence of a mercury compound on the setts decreased the efficiency of the plants growing from them in the production of tuber tissue—an effect opposite to that of zinc oxide.

The value for treatment 1 was below those for plants of similar leaf area in all other treatments, presumably because the setts were derived from an early-maturing clone of Up-to-Date.

The ratings for the condition of the setts, plotted against leaf area below the estimates of yield in Fig. 3, provide an explanation for the rising curve of yield on leaf area within each set of three treatments. With two exceptions, the sett ratings form a well graded series, falling as the leaf area and yield rise. The sett rating of treatment 1 is abnormally placed because of the origin of the setts. Treatment 3 has a relatively high value for sett rating where a lower one would be expected, but this is insufficient to nullify the general trend.

The indirect effect of treatment on yield may be summarized as follows:—The condition of the setts was modified by treatment. The plants of those treatments in which the setts remained sound and turgid grew more rapidly and produced larger tops than those in which the setts were in poor condition and rotted prematurely. This difference, in turn, was reflected in yields, the larger haulms on the whole producing the greater yields. The simple relation between size and yield, however, was probably complicated by direct effects of treatment on the ratio between haulms and tubers; and both size and yield were subject to the effects of unequal germination induced by treatment.

Even through this screen of disturbing influences it is possible to demonstrate associations between sett ratings and leaf area and sett ratings and yield. Using the 26 pairs of intra-block treatment means, a correlation coefficient of -0.4101 was calculated for sett rating and leaf area on January 23, and a coefficient of -0.4121 for sett rating and yield. Exclusion of the values for treatment 1, which were not comparable with those for other treatments, would have raised these coefficients to higher values.

9. Discussion

In this and the following paper, the results of a single field trial on the use of zinc oxide as a disinfectant for cut potato setts are examined in detail. The discovery that zinc oxide might be used to protect the cut surfaces of potato tubers is recent (Bald, 1943a). The experiment described here was designed to give a wide range of preliminary information by the collection of parallel data on disease incidence and on the growth of potato plants from treated and untreated setts. The results have been sought by comparison and inference, not by the repetition of experiments and the elimination of unwanted variables. The object has been to outline problems rather than find definitive answers.

The effects of zinc oxide on the condition of the setts and on growth and yield hold a promise of usefulness that warrants further work on its use as a protector of cut potato setts. Given conditions that normally favour suberization on the cut surfaces of tubers, zinc oxide is not toxic to tuber tissue, rather it favours suberization. It is probably fungistatic rather than directly toxic to fungi, and so weighs the balance in favour of the production of sound and healthy setts. It is not likely to compensate for careless methods of preparing cut setts for planting, but it is likely to enhance the efficiency of good methods.

Of the thirteen treatments in the trial, number 7 was the best in its effect on the condition of the sett, growth, and yield. The setts were cut, dipped in zinc oxide suspension, and maintained under humid conditions until planting. This treatment might in practice be more effectively applied, not to infected but to healthy setts. The setts might be cut directly into the zinc oxide suspension without the cut surfaces making contact with the skin of other setts until they were coated with zinc oxide. Also the cutting knife might be regularly dipped into the zinc oxide suspension.

The method of dipping whole tubers in a mercury compound before the setts were cut was not so effective as the best zinc oxide dip. The use of zinc oxide-DDT dust on the whole tubers before the setts were cut gave results similar to those of the corresponding mercury treatment. A more suitable filler than crude pyrophyllite might be found for making up the dust, or zinc oxide-DDT dust without a filler might be used. If control both of infestation with the potato moth and rotting of seed tubers in storage were wanted, as well as protection of the setts, such a dust might be very valuable.

The direct effect of zinc oxide on the growth of treated plants, suggested in Fig. 3, might result from a deficiency of zinc in the soil where the trial was conducted. In other deficient soils, the use of zinc oxide should increase the efficiency of the potato plant for the production of tubers. The direct effect of treatment would not be expected in soils containing plenty of available zinc.

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The Freezing Point of Soil Water in Relation to the Permanent Wilting Percentage

By C. G. Gurr[*]

Summary.

The relation between permanent wilting percentage and freezing point depression of soil water was investigated for eight Australian soils, and the results compared with those of other workers.

When the conditions of freezing were suitably controlled, the water content of soil corresponding to a depression of 1·5°C. was found to give a close approximation to the permanent wilting percentage.

The direct method of determining the permanent wilting percentage of soils, as described by Marshall and Williams (1942), requires a period of several weeks in which to grow plants in soil samples and allow them to wilt. An indirect determination can be made through the measurement of the depression of the freezing point of water in the soil. This method has not so far been used with Australian soils, and the present investigation was designed for the purpose of ascertaining the usefulness or otherwise of the method in the case of local soils.

The work of Schofield and Da Costa (1938), and of Bodman and Day (1943), on the energy relations of soil water as determined by the freezing point shows that approximately the same depression is found at the permanent wilting percentage in all soils. This is because freezing point depressions give a measure of the energy with which soil water is held, and the permanent wilting percentage corresponds to a definite value of this energy. This value may be calculated in terms of the free energy of escape of soil water, termed moisture potential, by the equation—

$$\text{moisture potential} = \frac{L\,T}{T_0} \text{ ergs/gram of water,}$$

where L = latent heat of fusion of water $(- 3 \cdot 336 \times 10^9 \text{ ergs/g.})$

$\qquad T$ = freezing point depression of water in soil (°C.)

$\qquad T_0$ = freezing point of pure water (273·18° absolute).

This quantity is numerically equal to an osmotic pressure of $\frac{L\,T}{T_0}$ dynes/sq. cm. or $\frac{L\,T}{T_0 \times 1 \cdot 013 \times 10^6}$ atmospheres.

To test the value of this method, the two procedures of Schofield and Da Costa (1938) were tried. In procedure A the use of three freezing baths gives control over the amount of supercooling, and reduces the effect of further cooling while freezing is taking place. Procedure B is less accurate in that only one freezing bath, at a temperature of about — 3°C., is used.

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The heat capacity of the freezing tube containing moist soil and Beckmann thermometer is determined in terms of water-equivalent in both procedures. Then an estimate of the percentage of ice formed during freezing may be calculated from the relation—

$$\text{ice as percentage of oven-dry soil} = \left(\frac{100W}{LS} + \frac{M}{L}\right) \triangle T$$

where W — water equivalent of tube + dry soil + thermometer (grams)

S — dry weight of soil in tube (grams)

. $\triangle T$ — rise in temperature during freezing (°C.)

L — latent heat of fusion (calories/gram)

M — water content of soil on oven-dry basis (%).

This estimate in procedure B is too low, owing to cooling during freezing. Early results showed that consistent results could not be obtained, and procedure B was not used further.

The calculated percentage of ice formed is subtracted from the percentage water content of soil on the oven-dry basis, to give the true water content at the measured freezing point.

Freezing point depressions were plotted (Fig. 1) on a logarithmic scale against corrected water content for eight soils (Table 1). On the curve for each soil the value of the permanent wilting percentage, obtained by the direct method, was interpolated to find the freezing point depression corresponding to that percentage. Results, for procedure A, are summarized in Table 2.

TABLE 1.—DETAILS OF SOIL SAMPLES USED.

Sample No	Location	Depth.	Clay Content	Moisture Equivalent	Total Soluble Salts.	pH.
		in.	%	%	%	
9228	Ord River, W.A.	0–9	5·2	3·8	·005	6·7
6398	Waikerie, S.A. ..	0–17	4·2	4·09	·027	8·8
9218	Ord River, W.A.	0–6	12·8	14·3	·008	6·8
6272	Waite Institute, S.A.	0–4	17·5	20·6	·039	5·9
4537A	Curlwaa, N.S.W.	0–11	33·6	21·0	·021	7·7
3927	Merbein, Vic.	0–7	13·8	16·3	·535	8·6
9224	Ord River, W.A.	0–7	50·4	27·4	·020	8·1
4459	Atherton Tableland, Qld.	Surface	31·3	27·8	·102	8·3

TABLE 2.—FREEZING POINT DEPRESSIONS AT THE PERMANENT WILTING PERCENTAGE.

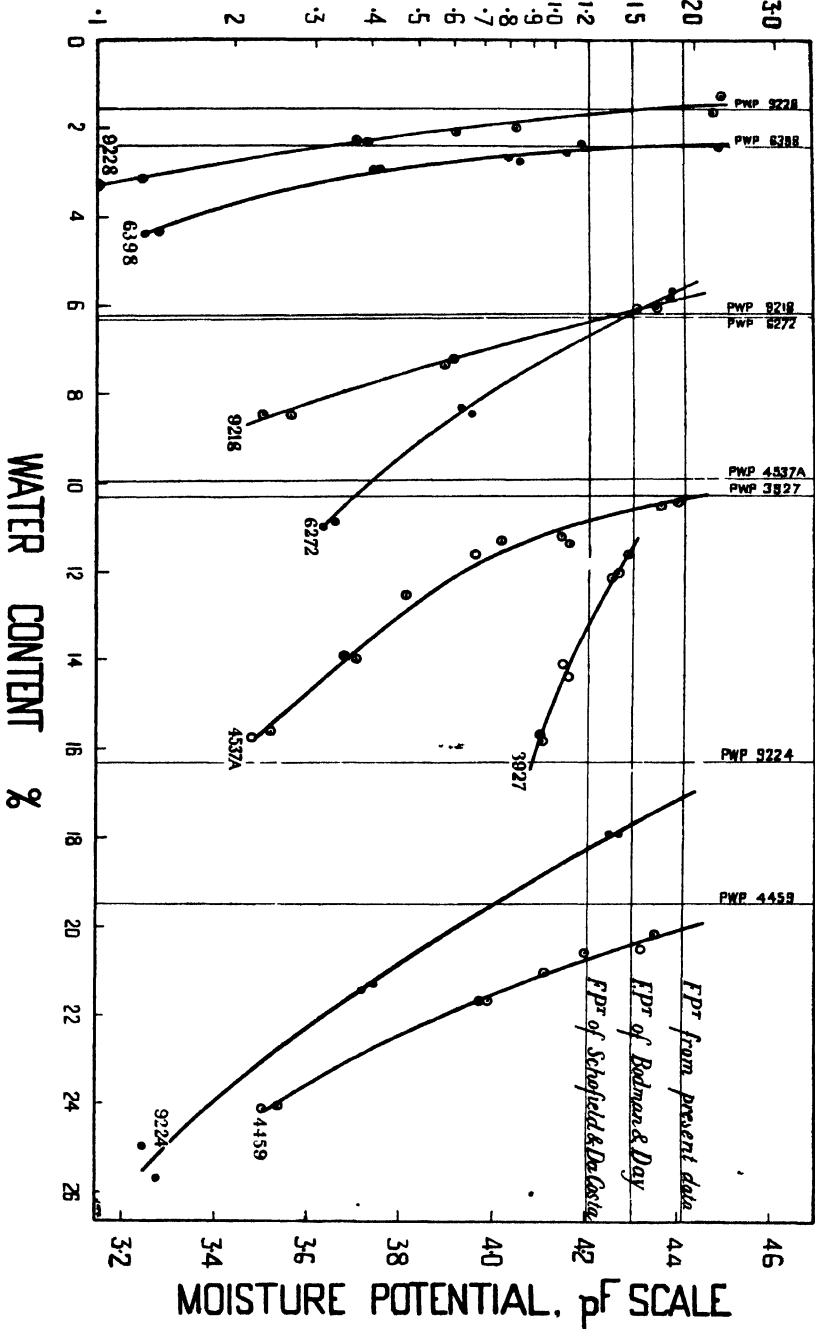| Soil No.				Permanent Wilting Percentage (Direct Method)	Depression of Freezing Point (Method A).
					°C.
9228	..	..	..	1·62	1·4
6398	..	..	..	2·41	1·5
9218	..	..	..	6·24	1·4
6272	..	..	..	6·32	1·4
4537A	..	..	..	9·95	3·1
3927	..	..	..	10·27	1·9
9224	..	..	..	16·33	2·7
4459	..	..	..	19·44	3·1

FIG. 1. The relation between depression of freezing point and water content for eight soils. The permanent wilting percentage (P.W.P.) determined by the direct method is also shown for each soil.

As can be seen in Fig. 1, the freezing point depressions given in Table 1 were in some cases obtained by extrapolation, owing to the difficulty in finding freezing points below about — 2°C. From Table 1 it appears that increasing values of permanent wilting percentage give greater values of depression of the freezing point. However, the data are not sufficient to be conclusive, and this trend is not shown in the results of other workers.

The geometric mean value for freezing point depressions given in Table 1 is 1·9°C. Schofield and Da Costa (1938) give 1·2°C., and Bodman and Day (1943), using a thermocouple method, give 1·5°C. as mean values. In terms of moisture potential at 0°C., these figures correspond to — 23·2 $\times$ 10⁶, — 18·3 $\times$ 10⁶, and — 14·7 $\times$ 10⁶ ergs/ gram of water respectively, or as equivalent osmotic pressure, 22·9 18·1, and 14·5 atmospheres respectively. Corresponding pF values (Schofield and Da Costa, 1938) are 4·4, 4·3, and 4·2.

Since the curves of freezing point depressions against water content have in general a steep slope in the region of the permanent wilting percentage, the variation in these mean figures represent only small differences in water content. Consequently, Bodman and Day's figure of 1·5°C., which is approximately the mean of these three figures, will give with reasonable accuracy the permanent wilting percentage of the eight soils used in the present comparison (Table 3).

TABLE 3.—COMPARISON BETWEEN PERMANENT WILTING PERCENTAGE AND WATER CONTENT CORRESPONDING TO FREEZING POINT DEPRESSIONS OF 1·9, 1·5, AND 1·2°C.

Soil Number	Permanent Wilting Percentage (Direct Method)	Water Contents Corresponding to Depressions of —		
		1·9°C.	1·5°C.	1·2°C.
	%	%	%	%
9228	1·62	1·5	1·6	1·7
6398	2·41	2·3	2·4	2·5
9218	6·24	5·9	6·2	6·4
6272	6·32	5·6	6·2	6·7
4537A	9·95	10·3	10·6	10·8
3927	10·27	10·3	11·5	12·8
9224	16·33	17·1	17·7	18·2
4459	19·44	20·0	20·3	20·5

The influence of salt concentration does not appear to alter the relation. For soil No: 3927 with its high salt content of 0·5 per cent., the curve (Fig. 1) is flattened; but the error in taking the water content at 1·5°C. depression as the permanent wilting percentage is only slightly greater than for the other soils.

Conclusions

The investigation confirms the experience of previous workers that the freezing point method offers a reasonably accurate means of determining the permanent wilting percentage of soils indirectly.

It is necessary for best results that three freezing baths be used in determining the freezing point of soil water, and that heat capacity be determined for estimation of ice formation.

A freezing point depression of 1·5°C. is a suitable basis for assessing the water content corresponding to the permanent wilting percentage.

Acknowledgment

The author wishes to thank Dr. T. J. Marshall for suggestions in preparing this paper.

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A Spectrochemical Survey of Some Phosphate Rocks and Superphosphates ˙

1. Qualitative

By A. C. Oertel, M.Sc.,* and H. C. T. Stace, B.Sc.*

Summary.

Fifteen samples of phosphate rock and ten samples of superphosphate have been examined spectrochemically. In addition to the major constituents, the presence of 23 other elements is noted with a maximum of 20 in any one sample.

Molybdenum was found in samples of phosphate rock from Egypt, Algeria, and Florida, but not in samples from Nauru and Ocean Island. As supplies of phosphate rock from these latter sources were cut off during the war, the war-time superphosphate contained more molybdenum than the pre-war quality.

Nickel was noted in all samples except those from Ocean Island and Nauru.

Cobalt was found only in samples from Florida, the low-grade phosphate rocks from South Australia, and two superphosphates.

The presence of some of these trace elements should be considered in the interpretation of any field experiments carried out with war-time superphosphate.

1. Introduction

Observations by Professor H. C. Trumble of the Waite Agricultural Research Institute on the apparent differences in response of pastures on Kangaroo Island to pre-war and war-time superphosphates, led to a detailed spectrochemical survey of 29 samples of phosphate rocks, superphosphates, and acid neutralizers. Several investigators (1–8) have examined mixed fertilizers, phosphate rocks, superphosphates, &c., by chemical and physico-chemical means for the " trace " plant nutrients.

Walkley (1) has determined zinc, cadmium, copper, and manganese in Nauru and Ocean Island phosphates and superphosphates manufactured from these sources.

Jacob *et al.* (2) in a survey of the phosphate resources of the United States of America made determinations for magnesium, titanium, sodium, manganese, potassium, &c., while Ballard (8) in a spectrochemical survey of twelve samples of fertilizers, amongst which were two phosphate rocks and two superphosphates, recorded the presence of 21 of the arc-sensitive elements.

This appears to be the first spectrochemical survey of a wide range of phosphates and superphosphates for those constituents which may be detected in the direct-current arc.

2. Experimental

Table 1 gives details of the samples examined. The samples as obtained were either finely divided or in small lumps. In the former case a sub-sample of from 1 to 2 grams in weight was ground in an agate mortar to secure homogeneity; in the latter case a larger

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* An officer of the Division of Soils.

sub-sample was finely ground in a porcelain mortar, and then the smaller sub-sample was mixed in the agate mortar. Duplicate samples of from 100 to 150 mg. were taken from each sub-sample, and each sample loaded into the cavity of a tared graphite electrode. These large samples were used to reduce the error caused by heterogeneity and to lessen the likelihood of missing volatile components present at low concentrations.

A Hilger Automatic Large Quartz Spectrograph (E 492) was used to record the spectrograms. The slit width used was 10 microns. The samples were arced, using a current of 15 amperes, an arc length of 4 mm., and an exposure of 60 seconds. Ilford Long-Range Spectrum plates were used for the wave-length range from 8000A–3200A and Kodak Process plates for the ranges 3500A–2460A and 2900A–2240A. The last range was photographed for selected samples only, in order to verify the presence of cadmium by the line Cd. 2288A.

To increase the range of concentrations which could be recorded at suitable densities on the plates, the spectrograms were stepped by using a rotating stepped sector of ratio 4. Each plate carried an iron arc spectrogram, stepped by a rotating stepped sector of ratio 2. That spectrogram was used to determine the H and D curve of the plate for each wave length of interest. The plates were developed for three minutes in 1: 2 metol-hydroquinone developer.

3. Discussion

A complete qualitative analysis, subject to the limitation imposed by the wave-length ranges used, was made of each sample. The results are recorded in Table 1. In that table, M denotes a major component; h, m, and l that the element is present at a concentration of the order of parts per 100, parts per 10,000, and parts per 1,000,000 respectively. The indication of the order of concentration is an estimate only, based on past experience, and may sometimes be in error for the less common elements.

The elements of agricultural interest include iron, manganese, copper, zinc, cobalt, molybdenum, and boron. Of these, it is seen that zinc and molybdenum are sometimes present in superphosphates at concentrations high enough to be important when the fertilizer is used in an experiment on a " deficient " soil. Figures by Walkley (1), who gives for Nauru and Ocean Island rocks 900 and 1,000 p.p.m. respectively, confirm these estimates. He has also shown that the amount present in a superphosphate depends upon the method of manufacture of the acid used in the production of the superphosphate. Superphosphate made with acid manufactured from zinc concentrates by the chamber process may contain as much as twice the amount of zinc as that produced with acids made from elementary sulphur or by the contact process.

In the case of molybdenum, it is of interest that it was detected in ten samples representing three localities: Kosseir, Egypt; Bona, Algeria; and Florida, U.S.A., listed in order of decreasing concentration.

While the amounts found were small—of the order of parts per million—they are perhaps not without significance. Anderson (9) has obtained field responses following the application of molybdenum trioxide at rates as little as 1/16 oz. per acre, which in a 2-cwt. per

TABLE 1.—SHOWING THE PHOSPHATE ROCKS, SUPERPHOSPHATES, AND ACID NEUTRALIZERS EXAMINED, WITH AN ESTIMATE OF THE ORDER OF CONCENTRATION FOR THE ELEMENTS DETECTED.

M = major component, h = parts per hundred, m = parts per thousand, l = parts per ten thousand, 1 = parts per million.

Description	Origin	Li	Na	K	Rb	Cu	Ag	Mg	Ca	Sr	Ba	Zn	Cd	Al	Si	Sn	Pb	P	Sb	Cr	Mo	Mn	F	Fe	Co	Ni
Phosphate rock	Nauru	1	m	1	·	·	1	m	M	m	1	m	1	m	m	·	1	M	1	1	·	1	h	m	·	·
"	Ocean Island	1	m	m	·	·	1	m	M	m	·	m	1	m	m	·	1	M	1	1	·	1	h	m	·	1
"	Safi, Morocco	1	h	m	m	m	1	m	M	m	1	m	1	m	h	·	1	M	1	m	·	1	h	m	·	1
"	Morocco	1	h	m	·	·	1	m	M	m	1	m	1	m	h	·	1	M	1	1	·	1	h	m	·	1
"	Bona, Algeria	1	h	1	·	m	1	m	M	m	1	1	1	m	h	·	1	M	1	m	1	1	m	h	·	1
"	Kosseir, Egypt	1	h	m	·	·	1	m	M	m	1	1	1	h	h	·	1	M	1	1	1	m	h	h	·	1
"	Florida, U.S.A.	1	h	m	·	·	·	m	M	m	1	1	1	h	h	·	1	M	·	1	1	1	m	h	·	1
"	Kapunda, South Australia	1	m	m	·	·	1	m	M	m	·	1	1	h	h	·	1	M	1	1	·	1	m	h	·	1
"	Hermitage, South Australia	1	m	m	·	·	·	m	M	m	m	·	1	h	h	·	·	M	·	·	1	m	m	m	·	·
"	Truro, South Australia	m	h	m	1	m	1	m	M	1	1	1	·	h	h	·	·	M	·	1	·	1	m	h	·	1
"	Ororoo, South Australia	m	m	m	1	m	1	m	M	m	1	m	·	h	h	1	·	M	·	1	·	m	m	m	·	1
Superphosphate	30 per cent. Makatea + 70 per cent. Kosseir	1	m	m	·	1	1	m	M	m	1	m	·	h	h	·	1	M	1	1	1	m	m	h	1	1
"	50 per cent Makatea + 40 per cent. Kosseir + 10 per cent. Kapunda	1	m	m	·	1	1	m	M	m	1	1	·	h	h	·	·	M	·	m	1	1	m	h	·	1
"	25 per cent Makatea + 75 per cent. Egyptian	1	m	m	·	1	1	m	M	m	1	1	·	m	h	·	1	M	·	1	1	1	m	m	·	1
"	45 per cent. Makatea + 55 per cent. Egyptian	1	m	m	·	m	1	m	M	m	1	1	1	m	h	·	1	M	·	m	1	1	h	m	·	1
"	100 per cent. Bona	1	h	m	·	·	1	m	M	m	1	1	1	m	1	·	1	M	1	1	1	m	m	h	·	1
"	100 per cent. Ocean Island	1	m	1	·	·	·	m	M	m	1	m	1	m	m	1	1	M	1	1	·	1	m	h	·	1
"	Ocean Island + Nauru	1	m	1	·	1	1	m	M	m	·	1	1	m	m	1	1	M	1	1	1	h	m	h	·	1
"	Serpentine Super from New Zealand	1	m	m	·	·	1	m	M	m	·	1	·	m	m	1	·	M	1	1	1	1	m	h	·	1
"	Special Super (granular)	1	m	m	·	·	·	h	M	m	1	1	1	m	h	·	1	M	1	1	1	m	m	h	·	1
Shellgrit	Angaston, South Australia	1	m	m	·	1	1	m	M	m	1	·	1	m	h	1	·	·	·	1	·	1	m	m	·	1
Marble	South Wallaroo, South Australia	1	h	h	·	1	·	m	M	m	·	·	1	h	m	1	·	·	1	1	·	m	·	m	1	1
Limestone sand	North Wallaroo, South Australia	1	h	h	·	1	·	h	M	m	1	·	1	h	h	·	·	·	·	1	1	1	·	m	1	1

NOTE.—B, Ti, V, were recorded in all spectrograms. These elements occur as impurities in the electrodes used, consequently no attempt was made at a quantitative estimate of them.

acre dressing of superphosphate is approximately 10 p.p.m. · Trumble and Ferres (10) have noted responses from subterranean clover in pots when using superphosphate prepared from Egyptian and Makatea rocks, compared with superphosphate from Ocean Island.

Cobalt was found in only eight samples, four of which were the low-grade phosphate rocks from South Australia. The others were the two from Florida, a Makatea—Egyptian superphosphate, and the serpentine superphosphate from New Zealand.

Other elements worthy of note are rubidium, which only occurred in the South Australian rocks, cadmium and antimony which occurred in eleven and fourteen of the samples respectively, and nickel which occurred in all the phosphate rocks and superphosphates, except those from Ocean Island, Nauru, and the Special superphosphate.

4. Acknowledgments

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Thanks are due to Mr. A. T. Hicks of Adelaide Chemical and Fertilizer Co. Ltd. and to Mr. C. A. Abbott of Cresco Fertilizers Ltd. who supplied the samples for the survey.

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The Solubility of Lead Fluoride in Aluminium Fluoride Solutions

*By T. R. Scott, M.Sc.**

The solubility of lead fluoride in solutions of aluminium fluoride, containing up to 17·5 g. AlF₃ in 100 g. of solution, has been determined at 25°C. Consideration has also been given to the variation of solubility with time of stirring, amount of excess hydrofluoric acid present, and temperature. The greatly increased solubility of lead fluoride is ascribed to depression of fluoride ion concentration by the formation of complex alumino-fluoride ions. The solutions formed in this way slowly deposit lead fluoaluminate. The coating of lead fluoride, which normally protects a lead surface against corrosion by hydrofluoric acid, is not formed in the presence of aluminium fluoride solutions and severe corrosion of lead vessels may therefore occur. Suggestions are made for other materials of construction which withstand corrosion by such solutions.

1. Introduction

In the course of experiments on the preparation of aluminium fluoride by the neutralization of hydrofluoric acid with aluminium hydroxide, it was found that the solid aluminium fluoride eventually recovered was grossly contaminated with lead fluoride derived from the lead vessels in which the operations had been conducted. In the presence of hydrofluoric acid such vessels acquire a protective layer of lead fluoride; solutions of aluminium fluoride, however, dissolve this layer and the freshly-exposed lead surface may undergo further corrosion.

Since lead is commonly employed as a material of construction in plants where hydrofluoric acid is used, the solubility of lead fluoride in solutions of aluminium fluoride was investigated. A search of the literature has disclosed no quantitative work on this subject, though Fischer and Thiele (3) have commented on the considerable quantities of lead remaining in solution when acid solutions of aluminium fluoride are neutralized with lead carbonate.

From the solubility determinations it should be possible to decide if lead is ever suitable for use with aluminium fluoride solutions and to suggest other materials better able to withstand corrosion in such media.

2. Experimental Methods

The following factors were found to influence the solubility of lead fluoride in aluminium fluoride solutions:—

      (i) Time for which solutions were stirred.
      (ii) Concentration of aluminium fluoride.
      (iii) Acidity.
      (iv) Temperature.

For solubility determinations, a considerable excess of lead fluoride was stirred with aluminium fluoride solutions in monel beakers, using silver-plated copper stirrers. Under these conditions no contamination

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of the solutions by the metals employed could be detected. In the majority of experiments, solutions were maintained at a temperature of $25 \cdot 0 \pm 0 \cdot 1^\circ$C. in a thermostatted bath, although results obtained subsequently showed that accurate temperature control was not necessary.

Solutions of aluminium fluoride were prepared in rubber-lined vessels from Laboratory Reagent aluminium sheet and A.R. hydrofluoric acid. By completing the neutralization overnight with pure aluminium foil, it was possible to obtain solutions having a pH value of at least $4 \cdot 0$. The lead fluoride used was of Laboratory Reagent grade, the pH value of aqueous solutions being $5 \cdot 3$ at 25°C.

The lead content of the filtered solutions was determined by the addition of an excess of sulphuric acid, precipitation of lead sulphate being quantitative in the presence of N-sulphuric acid. The precipitate was washed with dilute sulphuric acid and aqueous ethanol (50 per cent. water) and dried to constant weight at 135°C.

Fluorine was eliminated from a suitable aliquot of the filtrate by evaporation to dryness and digestion with concentrated sulphuric acid, after which the aluminium content was determined by precipitation with 8–hydroxyquinoline, as described by Mellan (5).

Solutions of aluminium fluoride are metastable at the concentrations used in these experiments and slowly deposit β–$AlF_3 . 3H_2O$(2). No useful purpose could therefore have been served by analysis of the solid residues obtained in the stirring runs, since equilibrium conditions were not attained. The aim of the investigation was rather to determine the maximum amounts of lead fluoride dissolved under varied conditions, and this maximum was reached quite rapidly, as will be shown later.

3. Experimental Results

(i) *Time of Stirring.*

Preliminary stirring runs, to determine the time required to reach the maximum solubility of lead fluoride, disclosed that the solutions are frequently supersaturated with lead fluoaluminates, which slowly deposit on the sides and bottom of the containing vessel to form an adherent coating. In contrast to pure concentrated solutions of aluminium fluoride, which require nine months to come to equilibrium at 25°C. (2), the solutions containing lead fluoride deposit the major part of the lead as fluoaluminates within 24 hours, the actual time depending upon the concentration of the solutions. The maximum solubility for lead fluoride is reached in 60–80 minutes for solutions containing more than 10 per cent. aluminium fluoride, but the time is decreased to fifteen minutes or less with more dilute solutions. At each concentration of aluminium fluoride, therefore, samples were drawn from the vessels at frequent intervals, and the maximum solubility chosen from the analytical results. Under such circumstances, it is possible that the absolute maximum value was not always obtained, and this source of error is apparent in Fig. 1; from the same figure, however, it is evident that the error is not serious since the points all lie close to a smooth curve of best fit.

Reproducible results could be obtained only if the solutions were thoroughly stirred from the moment of introduction of the lead fluoride. This prevented caking of the lead fluoride until the maximum amount had been dissolved, after which stage the onset of caking gave an indication that the solubility was decreasing owing to deposition of lead fluoaluminates.
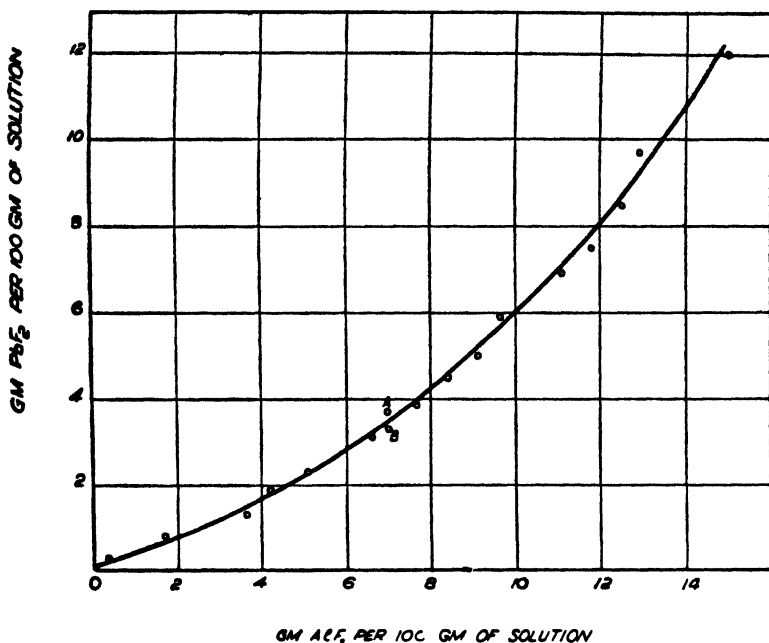
FIG 1 —Solubility of lead fluoride in aluminium fluoride solutions at 25°C

(ii) *Concentration of Aluminium Fluoride.*

Results are shown graphically in Fig. 1. The most concentrated solutions obtained were found to contain 12 per cent. of lead fluoride, compared with the low value of $0 \cdot 064$ per cent. for lead fluoride in water.

The concentration of lead fluoride increases more rapidly than that of aluminium fluoride; hence a precipitate is formed when concentrated solutions are diluted with water. Analysis of this material, after filtering and washing with small amounts of water and alcohol, showed the molar ratio $AlF_3 : PbF_2$ to be $0 \cdot 97 : 1 \cdot 00$, from which it appears that the precipitate has the empirical formula $PbAlF_5$. If the precipitate is washed many times with water, however, the molar ratio $AlF_3 : PbF_2$ in the dried solid is decreased considerably, indicating that the compound is incongruently soluble in water. For this reason, the compound could not be leached from melts of aluminium and lead fluorides having the composition of $PbAlF_5$, although the amounts of lead and aluminium in the resulting solutions were considerably greater than could be found in saturated solutions of the simple anhydrous fluorides.

(iii) *Acidity of Solutions.*

By adding hydrofluoric acid to aluminium fluoride solutions, the solubility of lead fluoride can be greatly decreased. The effect is illustrated in Fig. 2 for solutions which all contain the same concentration of aluminium fluoride (7·1 per cent.).

In the presence of relatively small amounts of acid, however, the maximum solubility is not significantly different from that for completely neutralized solutions. The values for points A (pH 2·75) and B (pH 1·87) in Fig. 2 both lie reasonably close to the best-fit solubility curve shown in Fig. 1, although at point B the solution contains 0·13 moles of hydrofluoric acid per mole of aluminium fluoride. There is little doubt that the solubility does decrease with decreasing pH even in such weakly acid solutions, but the errors inherent in the determination of the true maximum solubility tend to mask the effect.
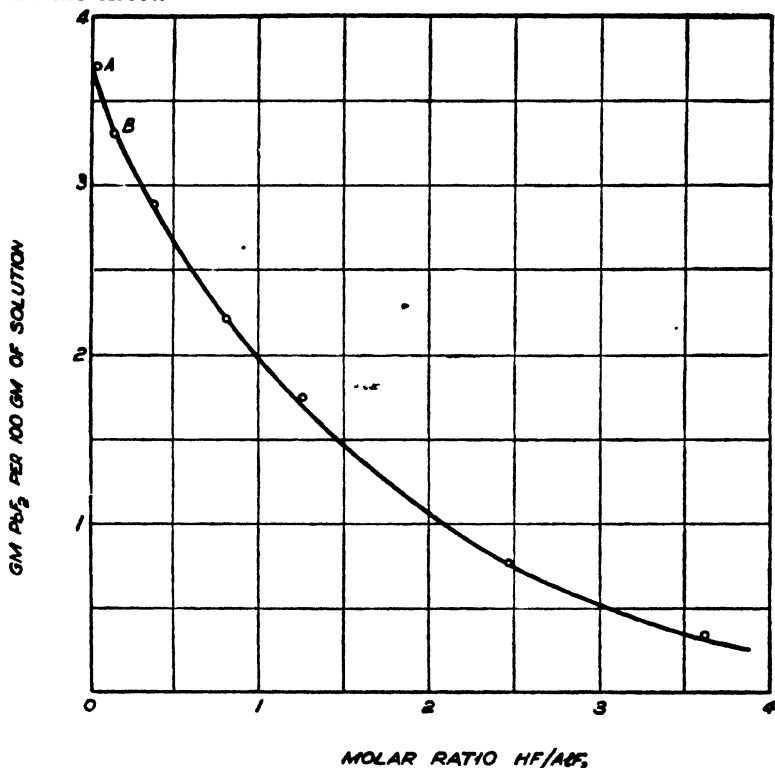
FIG. 2.—Effect of hydrofluoric acid on the solubility of lead fluoride in 7·1 per cent. aluminium fluoride solution.

By using a mixture containing two parts by weight of fluosilicic acid to fifteen parts of hydrofluoric acid, instead of pure hydrofluoric acid, the solubility of lead fluoride is increased by approximately 25 per cent. Such a mixture contains the two acids in the proportion commonly found in commercial grades of hydrofluoric acid.

Greatly *increased* solubilities occur when other acids, such as fluosilicic, perchloric, and acetic acids, are substituted for hydrofluoric acid in the above experiments.

(iv) *Temperature of Solutions.*

At high temperatures, the maximum solubility is reached more rapidly than at room temperature, and the aluminium fluoride solutions tend to deposit β-AlF_3 . $3H_2O$ more readily. Solubility determinations are consequently difficult to perform satisfactorily. Comparative figures are given, however, in Table 1, for four points along the solubility curve, the values at 25°C. being taken from the curve in Fig. 1. These high-temperature determinations were done within the temperature range 90–98°C., and suffice to show that the temperature coefficient of solubility is relatively small.

TABLE 1.—EFFECT OF TEMPERATURE ON SOLUBILITY.

AlF_3 g /100 g of Solution	PbF_2 g /100 g. of Solution	
	25°C	90°C.
13·0	9·4	10·2
11·3	7·2	8·7
7·2	3·6	4·3
3·9	1·7	2·1

4. Discussion

Since the solubility product of lead fluoride is $7 \cdot 1 \times 10^{-8}$, it appears that the greatly enhanced solubility in aluminium fluoride solutions is due to a reduction in the concentration of lead or fluoride ions. This reduction may be brought about in three ways:—

(a) Formation of associated (non-ionized) molecules of lead fluoaluminate.

(b) Formation of complex ions of the type PbF_i^-.

(c) Formation of complex ions such as $AlF_6^{\equiv}$.

Of these suggestions, the first is improbable and no evidence has been adduced for the existence of PbF_4 anions. Lead fluoride is almost insoluble in hydrofluoric acid, and is immediately precipitated by the addition of lead nitrate to this acid. From solutions of lead fluoride in aluminium fluoride, lead sulphate is completely precipitated by the addition of sulphuric acid and the metal is rapidly deposited in spongy form on less noble metals placed in the solutions.

There is abundant evidence (1), however, that solutions of aluminium fluoride may contain complex ions of the type AlF^{++}, AlF_2^+, AlF_4^-, $AlF_5^=$ and $AlF_6^{\equiv}$. The existence of the complex cations is demonstrated by the fact that aluminium hydroxide is not precipitated at pH 4·1 when ammonia is added to these solutions. Instead, precipitation is delayed until the pH reaches a value of five or higher, and the precipitates apparently consist of aluminium oxyfluorides, or hydroxyfluorides, such as $AlF_2(OH)$. The complex anions are found in the various alkali and ammonium fluoaluminates which are precipitated on the addition of the appropriate compounds. The possibility

that " multiple core " ions, such as $(Al_3F_{14})^{5-}$, may be present has not been overlooked, but it is simpler to dismiss them from discussion, which may be done without prejudice to the suggestions put forward.

When lead fluoride is added to these solutions, the following reactions, involving the complex ions originally present, can be envisaged: —

$$F^- + AlF^{++} \rightleftharpoons AlF_2^+ \qquad \dots\dots\dots \text{(I)}$$

$$F^- + AlF_2^+ \rightleftharpoons {}^-AlF_3 \qquad \dots\dots\dots \text{(II)}$$

$$F^- + AlF_3 \rightleftharpoons AlF_4^- \qquad \dots\dots\dots \text{(III)}$$

$$F^- + AlF_4^- \rightleftharpoons AlF_5^= \qquad \dots\dots\dots \text{(IV)}$$

$$F^- + AlF_5^= \rightleftharpoons AlF_6^{\equiv-} \qquad \dots\dots\dots \text{(V)}$$

In this manner the fluoride ion concentration is held at a very low value and considerable concentrations of lead ions may exist in solution without the solubility product of lead fluoride being exceeded.

In solutions containing aluminium fluoride only, the various complex ions are present in definite equilibrium proportions, but with the introduction of lead and fluoride ions, these proportions are considerably changed. The delay in precipitation of lead fluoaluminates from such solutions may well be caused by a slow re-establishment of the equilibrium proportions of the complex ions. If it be assumed that one of the above reactions, such as reaction (I.), is predominant, high concentrations of lead ions may occur without the solubility product of lead fluoaluminates (such as $PbAlF_5$) being exceeded. With time, however, equilibrium between the complex ions will be re-established, with a consequent decrease in the concentration of AlF_2^+ ions and a proportionate increase in the concentration of ions such as AlF_5^- At some stage in this process the concentration of AlF_5^- ions will reach the level at which the solubility product of $PbAlF_5$ is exceeded and precipitation will commence. Support for this hypothesis is afforded by the observations of Brosset (1), who found that, with very dilute solutions of aluminium fluoride, as much as one hour may elapse before equilibrium is established between the various ions. Even if reaction (I.) is not predominant, this explanation still holds, provided only that reaction (IV.) is of minor importance and that $PbAlF_5$ is the least soluble of the various lead fluoaluminates which can be postulated.

Addition of acids to solutions of aluminium fluoride should increase the solubility of lead fluoride by depressing the fluorine ion concentration. Unequivocal results could not be obtained with nitric, fluosilicic, and perchloric acids, since lead fluoride is soluble in these acids in the absence of aluminium fluoride. A distinct increase in solubility was noted, however, when small amounts of fluosilicic acid were added to solutions already acidified with hydrofluoric acid (see above).

When hydrofluoric acid is present, the concentrations of all the complex ions, including $AlF_5^=$, are increased and less lead fluoride can be added before the concentration of AlF_5^- becomes sufficient to cause

precipitation of PbAlF$_6$. The solubility of lead fluoride, therefore, shows a marked decrease with increasing concentration of hydrofluoric acid (Fig. 2). It is evident that the stage must eventually be reached where the supply of complex ions is inadequate to react completely with added fluorine. Even when the solution has the empirical composition of H$_3$AlF$_6$, however, the maximum amount of lead fluoride dissolved is appreciably greater than that dissolved by water or pure hydrofluoric acid.

In the light of the above explanation, it might be expected that other fluorides having very small solubilities would be much more soluble in aluminium fluoride solutions, but no increase in solubility was noted with the fluorides of barium, thorium, and magnesium (solubilities 0·17[10], 0·020[25], 0·0076[18] respectively; cf. PbF$_2$ 0·064[20]). The critical factor is apparently the solubility of the corresponding fluoaluminate; if this be less soluble than the fluoride, as occurs with barium, no increase in the concentration of barium will be observed in the presence of aluminium fluoride solutions. Lead fluoaluminate, on the other hand, is more soluble than lead fluoride. The solubility of magnesium fluoride is so slight that it is difficult to check the solubility of the fluoaluminate, but the latter compound is slowly precipitated when magnesium sulphate is added to aluminium fluoride solution. Thorium fluoride is precipitated from solutions of aluminium fluoride on the addition of thorium nitrate, but it is doubtful whether a fluoaluminate of thorium exists at all.

When small quantities of thorium nitrate are added to a clear solution of lead fluoride in aluminium fluoride, thorium fluoride is precipitated. From calculations based on the solubilities given above, however, lead fluoride might be expected to precipitate just as readily, since the concentration of lead ions is very much greater than that of thorium ions. This behaviour of thorium fluoride may be explained on the assumption that the quoted solubility is too great, a possibility which is further indicated by the following facts. When lead nitrate is added to an excess of hydrofluoric acid, a heavy precipitate of lead fluoride is obtained, but with added nitric acid the precipitate is quickly dissolved. This effect is undoubtedly due to depression of the fluorine ion concentration below the value required to cause precipitation of lead fluoride, yet when thorium nitrate is added an immediate precipitate of thorium fluoride is formed. It is, of course, conceivable that sufficient PbF$^+$ ions are present to reduce the concentration of lead and fluorine ions in these solutions, but it is difficult to assess the effect of this upon the solubility of lead fluoride, since enough is not known about the value of the solubility product [PbF$^+$] [F$^-$].

5. Applications

From the experimental results, it is evident that lead is unsuitable for use in the presence of aluminium fluoride solutions. Although the contamination of these solutions is very much reduced by the addition of hydrofluoric acid, the quantity of lead fluoride dissolved is still sufficient to give 0·2—0·3 per cent PbF$_2$ in the recovered aluminium fluoride, while the accompanying corrosion of the lead vessels is well beyond the limits which can be tolerated. The phenomenon has even been observed in the preparation of cryolite by

the neutralization of trisodium aluminate solutions with hydrofluoric acid in lead vessels, the resulting cryolite containing up to $0 \cdot 4$ per cent. lead fluoride.

Attempts have been made to overcome this corrosion by developing, on the lead surfaces, protective films of less soluble lead salts, such as the sulphate and chromate, but in no instance could sufficiently coherent and impervious films be obtained.

It is probable that this severe corrosion of lead vessels is not confined to solutions of aluminium fluoride, but may occur in any solution containing a variety of complex metallo-fluoride ions. The essential requirement appears to be that reactions of the type, $AlF_4^- + F^- \rightleftharpoons AlF_5^=$ and $AlF^{++} + F^- \rightleftharpoons AlF_2^+$, should be possible and many fluorides of the metals of higher valency, such as iron, chromium, vanadium, zirconium, and tantalum, comply with this condition. Support for this belief has been afforded by the observation (4) that, when zirconium phosphate is dissolved in hydrofluoric acid, using a lead vessel, considerable quantities of lead are found in solution.

In many operations involving the use of hydrofluoric acid, it is thus advisable to use vessels constructed of metals other than lead. It is possible, of course, to use synthetic resins, wood, graphite, &c., but there are occasions where a metal is more convenient and suitable. In such instances, the choice lies between metals which acquire an insoluble protecting fluoride film and those which are highly cathodic in the presence of acids. Dow metal (an alloy of magnesium and aluminium) belongs to the first group, and experiments described above have shown that the magnesium fluoride film is practically insoluble in aluminium fluoride solutions. Should the film be damaged, however, rapid corrosion of the metal may occur, and the subsequent re-establishment of a continuous and impervious film may be impracticable. To the second group belong metals such as copper, monel, and silver, which show little or no reaction with aqueous hydrofluoric acid, although the respective fluorides are quite soluble. Of these, silver is the most highly cathodic, but monel is probably the most suitable for everyday use, having the advantage over copper that joints in monel apparatus may be welded with monel scrap. No corrosion of monel ware has been observed in any of the experiments described in the course of this paper.

6. References

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Theory of the Bourdon Tube

By Elizabeth H. Mann, B.A.*

Summary.

A paper by H. Lorenz on the theory of the Bourdon tube pressure gauge has been reviewed and an alternative approach developed. This is only a first order approximation, and gives best results for flat thin-walled tubes.

Symbols

a .. Length of major semi-axis of tube cross-section (parallel with ZZ axis)

b .. Length of minor semi-axis of tube cross-section

$2h$ .. Wall thickness (Lorenz uses h)

r_0, R .. Radius of curvature of the tube (measured at major axis of symmetry)

r .. Radius of curvature at any point on tube cross-section about ZZ axis

χ .. Angle subtended at ZZ axis by arc of tube

p .. Pressure difference in tube

σ' .. Tangential or ring stress

σ'' .. Transverse or meridian stress

ZZ .. Axis of curvature of the tube

Φ .. Angle formed by the ZZ axis and the tangent at any point on the meridian line of the tube cross-section

θ .. Polar angle of the ellipse

$\overline{M'}$ .. Bending moment along a meridional boundary of tube wall

$\overline{M''}$ .. Bending moment along a tangential boundary of tube wall

τ .. Normal shear along a tangential boundary of tube wall

τ' .. Normal shear along a meridional boundary of tube wall

ρ' .. Radius of curvature of a principal section normal to meridian curve

ρ'' .. Radius of curvature of meridian curve of cross-section

κ .. Change in principal curvature $\dfrac{\cos \Phi}{r}$

κ' .. Change in principal curvature ρ''

Suffixes 1 and 2 are used to denote initial and final states respectively.

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1. Introduction

This investigation commenced as a review of an article by H Lorenz on the theory of the Bourdon tube pressure gauge[1] This led to the study of other papers on the same subject and finally to a fresh attempt to solve the problem

2. Discussion

The article by Lorenz[1] will be considered first His problem is to determine the stresses and deflection of a curved tube of elliptical cross-section under an internal pressure p The tube is curved so that its axis forms an arc of a circle subtending an angle X at the centre The major axis of the ellipse generates a cylinder about the ZZ axis (see Fig 1)
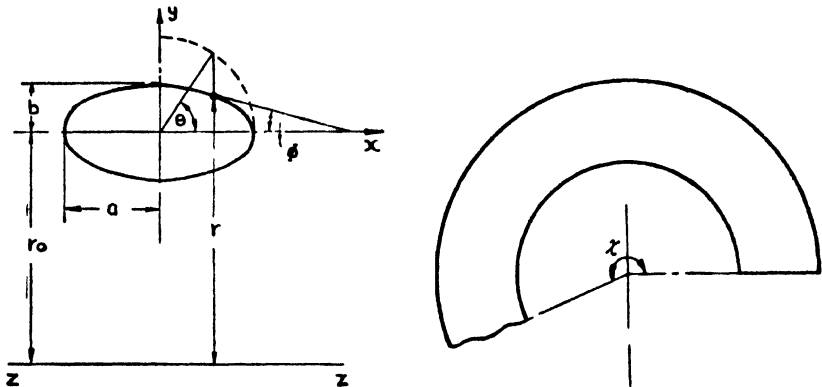
Fig 1

The tube is closed by soldered-on end pieces When stressed these might well cause some outward bending of the walls of the tube, but experience shows that such bending moments die away rapidly at a short distance from the end Thus there is justification for the omission of these terms and the assumption that the stress system does not vary with X However, Lorenz goes further than this and assumes axial symmetry, which enables him to use equations of equilibrium previously derived for a closed ring This involves a contradiction, since the method is to be used to calculate the change in angle X, implying a tangential displacement which is impossible under axial symmetry.
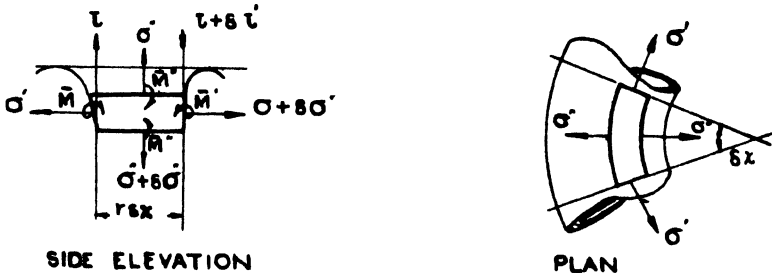
SIDE ELEVATION   PLAN

Fig 2

Moreover, the simplification made in neglecting the bending due to end effects is no reason for omitting $\overline{M}'$ altogether, as Lorenz has done (see Fig 2) His argument is this. from consideration of the equilibrium of

an element of the tube bounded by lines of constant r and X, and neglecting the quantities τ' and $\overline{M'}$, three equations are derived connecting the tensions σ', σ'', the shear τ, and bending moment M'' with the pressure p.

These are

$$\sigma'' = \frac{p}{h}\,\frac{r^2 - r_0^2}{2r\,\cos\,\Phi} + \tau\,\tan\,\Phi$$

$$\sigma' = \frac{p\rho'}{h}\left(1 - \frac{r^2 - r_0^2}{2r\rho''\,\cos\,\Phi}\right) - \frac{1}{\rho''}\,\frac{\partial}{\partial\Phi}\left(\frac{\tau r}{\cos\,\Phi}\right)$$

$$\frac{\partial\overline{M}''}{\partial s} = h\tau r X$$

where $ds = \rho'd\Phi$ is the element of arc of the meridian curve, and ρ', ρ'' are radii of curvature.

To solve these τr is replaced by $A \cos\,\Phi \sin\,\Phi$ and the constant A found from the condition that the strain energy is to be a minimum.

The resultant bending moment acting on the tube is, according to Lorenz, given by

$$M' = \int (r - r_0)\,\sigma'\,hds.$$

The strain energy L may then be expressed as a function of p, and the relative rotation $\triangle X$ of the ends of the tube found from the relation

$$\triangle X = \frac{\partial L}{\partial M'} = \frac{\partial L}{\partial p}\,\frac{\partial p}{\partial M'}.$$

Taking the points in order, the more exact equations

$$\frac{\partial}{\partial r}\,(\sigma''\,r\,\cos\,\Phi) - \frac{pr}{h} - \frac{\partial(\tau r\,\sin\,\Phi)}{\partial r} + \frac{\partial\tau'}{\partial X} = 0$$

$$\sigma' = \frac{p\rho'}{h} - \frac{1}{\rho''\,\cos\,\Phi}\,\frac{\partial(\tau r)}{\partial\Phi} - \frac{\sigma''\rho'}{\rho''} - \frac{1}{\cos\,\Phi}\,\frac{\partial\tau'}{\partial X}$$

$$\frac{\partial(\sigma'\rho'')}{\partial X} = \rho''\tau'\,\cos\,\Phi$$

$$\frac{\partial(\overline{M}''r)}{\partial s} = h\tau r - \overline{M}'\,\sin\,\Phi$$

$$\frac{\partial\overline{M}'}{\partial X} = h\tau'r$$

reduce to those given by Lorenz on omitting τ' and integrating the first with respect to r. The last gives $\overline{M}'$ constant with respect to X but not necessarily zero. $\overline{M}''$ is the bending moment per unit length, so that

$$M'' = \overline{M}''rX$$

and Lorenz's third equation follows if $\overline{M}'$ is zero. Whether or not it can be zero will be discussed later.

The substitution of $A \cos \Phi \sin \Phi$ for τ is suggested by the assumption that the tube retains its doubly symmetrical cross-section, so that τ vanishes at $\Phi = \dfrac{n\pi}{2}$ ($n = 1, 2 \ldots$). This sort of simplification is justified by common practice, and the assumption itself is consistent with his general assumption of infinitesimal strain.

The next point, however, is more troublesome. If M' represents the resultant bending moment of the stresses about a point on the axis of the tube, this must be equal to the moment of the external forces about the same point. The latter is due to the pressure acting on the walls and closed end of the tube to one side of the point about which the moment is to be found. This moment is readily shown to be zero not only for a tube of elliptic or circular cross-section but for any tube the cross-section of which has an axis of symmetry parallel to the ZZ axis (App. I.). If then M' is to represent the resultant moment it must be identically zero, and $\dfrac{\partial p}{\partial M'}$ is meaningless.

On the other hand the resultant moment is in fact equal to

$$\int \left((r - r_0)\ \sigma'h - \overline{M'} \cos \Phi \right) ds$$

so that his M' may be non-zero if $\overline{M'}$ does not vanish. This is one of the main contradictions in the work, that $\overline{M'}$ is neglected in the governing equations, while equilibrium considerations demand that it be sufficiently large to compensate the moment about the centroid due to the σ'.

Finally it is hard to justify the step

$$\triangle \chi = \frac{\partial L}{\partial M'}$$

for the rotation at the end; for the governing equations do not apply at the ends, M' is not the resultant couple acting on the tube, and if it were it would have to be zero at the free end. The latter in Lorenz's equations would make not only M' but p and all the stresses zero everywhere.

These considerations made the solution appear of doubtful value, and it was felt that some alternative should be found.

The articles by Case[2] and Beskin[3] on the bending of curved tubes were not applicable since in this case the resultant bending moment about a point on the axis of the tube is zero. (App. I.) Both replace a bending moment by a statically equivalent system of varying pressure within the tube, and at first it was thought that the reverse method might yield results.

Qualitative accounts of the Bourdon tube had been given by Hill[6], Worthington[7], and Greenhill[8] by the end of last century. They discussed some of the difficulties of the problem and described the most suitable dimensions for a tube of high sensitivity.

Lord Rayleigh[5] mentions the Bourdon tube as an example of Gauss's Theorem, that if extension is neglected the state of bending of a thin shell will be such that the product of the principal radii of curvature remains

constant. He points out, however, that, for an elliptical cross-section, pure bending cannot occur, and suggests replacing the two halves of the ellipse by symmetrical curves meeting at a finite angle.

Experimental results supporting the view that the deflection is largely due to inextensional bending are given by Wuest[9]. He calculates the path followed by the free end of the tube on the assumption that $(r\chi)$ has the same value before and after strain. He also deduces the locus of the instantaneous centre of curvature of the path, and verifies this experimentally. Reference is made to a paper by Strohmeyer[10], who discusses the Bourdon tube at length, describing the shearing effect between adjacent longitudinal strips. He suggests that the ideal cross-section for the gauge would be lip-shaped, not elliptic, to reduce this effect.

Sunatani[11] uses the theory of a straight tube under internal pressure by neglecting the curvature of short elements of the tube. In effect, he neglects longitudinal bending, which he justifies by showing that the resultant bending moment due to the pressure is zero (as in App. I.). Actually this moment is zero only about points on the axis of the tube, not in the walls. However, the effect of longitudinal bending is usually small compared with bending of the section, and his work is supported by experimental results. The deflection of the free end is calculated, shown to be largely dependent on the terms due to inextensional bending, and on the basis of these an improved mechanism devised to give a linear scale reading.

The problem of a tube of circular cross-section is discussed by Dean[4]. His tube forms a closed ring so is not really applicable to the Bourdon tube, but several closely analagous difficulties arise. He is led to the conclusion that the theory based on small displacements is not satisfactory, owing to the occurrence of large and approximately inextensional displacements which tend to make the circular cross-section oval.

The same difficulty arises with the Bourdon tube from the vanishing of the moment due to the pressure. The hypothesis that the displacements are small, implying that an element of the shell is in approximately the same position before and after strain, must be abandoned, and the difference between initial and final co-ordinates taken into account.

Under high pressure the elliptic cross-section will distort, tending to a circular shape, and causing a displacement away from the ZZ axis if $r > r_0$ and towards it if $r < r_0$. In consequence, different longitudinal strips have different centres of curvature, and if the angle χ remained the same the outer side of the tube would be in tension and the inner in compression. Equilibrium is attained by a change of both radius and angle causing the tube to straighten.

Another straightening effect is due to the extension of the tube caused by the pressure on the ends. Tangential extension of each element causes an increase in the radius of curvature and hence a small deflection, of the order of strain.

If the cross-section is circular it will not distort, but the increase in the radius will have a straightening effect on the tube. This will be of the same order of magnitude as the effect of longitudinal strain so both must be considered in the circular case.

If, however, the cross-section is fairly flat, with minor axis in the plane of bending, and the walls of the tube are thin, it is to be expected that the main effect is due to distortion towards the circular form. This problem seems to demand a treatment based on pure bending of a thin shell.

3. Approximate Solution

In this approach the difference between initial and final co-ordinates is no longer negligible. The suffix 1 indicates the initial state and suffix 2 the final. Lorenz's quantity r_0 is replaced by R_1 and R_2 respectively.

Pure bending only is considered, the effect of extension being neglected. Thus all lengths in the middle surface are supposed to remain the same after strain as they were before, although the form of the surface may alter considerably. For example, if the axis of the tube initially subtends an angle X_1 at the centre of a circle of radius R_1, and after strain subtends an angle X_2, then the new radius R_2 is given by

$$R_1 X_1 = R_2 X_2$$

In this way the final state may be expressed as a function of the initial state and only one other variable, that is, if the initial form of the tube and the final value of one dimension are known, the final state is determined. The variable chosen below is b_2, the final value of the minor semi-axis of the ellipse. The major axis can be expressed in terms of this from the condition that the length of the perimeter remain the same. Moreover, although it is not possible in the elliptic tube[5] for pure bending of the cross section to lead to pure bending longitudinally, yet for a fairly flat tube the extreme outer and inner parts of the shell will have most effect, and may be used to express X_2 as a function of b_2. For, if no extension occurs

$$(R_1 + b_1)X_1 = (R_2 + b_2)X_2$$
$$(R_1 - b_1)X_1 = (R_2 - b_2)X_2$$

Therefore

$$\frac{b_1}{b_2} = \frac{X_2}{X_1} = \frac{R_1}{R_2}$$

Having expressed the dimensions of the strained tube as functions of b_2, it only remains to find b_2 as a function of the pressure in the tube and the elastic constants of the material. This is done by using the theorem that the total energy of the system must be stationary for equilibrium, requiring that the increase of strain energy in a small displacement from equilibrium is equal to the work done by the internal pressure. Solving this equation gives b_2 and hence the other unknowns.

This method has been applied to the elliptic tube and also to one of lip-shaped cross section as suggested by Strohmeyer[10]. Both involved elliptic integrals and it was found convenient to approximate to the solution by power series. It was not considered worth while to take many terms of the series since the solution is already incapable of accuracy due to neglecting extension and assuming the form of the final shape.

The elliptic case alone will be given in detail. The form taken for the lip-shaped cross section was given by

$$y = b \cos^2 \frac{\pi x}{2a} \; .$$

The length of the perimeter is then

$$s = 4 \int_0^a \sqrt{1 + \frac{\pi^2 b^2}{4a^2} \sin^2 \frac{\pi x}{a}} \; dx$$

$$= 4b \sqrt{1 + \frac{4}{\pi^2 b^2}} \int_0^{\frac{\pi}{2}} \sqrt{1 - \frac{1}{1 + \frac{4a^2}{\pi^2 b^2}} \sin^2 x} \; dx$$

This is expanded as a series in powers of $\dfrac{1}{1 + \dfrac{4a^2}{\pi^2 b^2}}$. The procedure

otherwise is similar to that in the elliptic case.

An ellipse of semi-axes a_1, b_1 is supposed to bend without extension to form an adjacent ellipse of semi-axes a_2, b_2. A point on the first ellipse moves to a neighbouring point on the second at the same distance along the curve from an extreme point, that is

$$s_1 = \int_0^{\theta_1} \sqrt{a_1^2 - (a_1^2 - b_1^2) \cos^2 \theta} \; d\theta$$

$$= \int_0^{\theta_2} \sqrt{a_2^2 - (a_2^2 - b_2^2) \cos^2 \theta} \; d\theta$$

$$= s_2 \qquad \qquad \cdot \quad \cdot \quad (1)$$

For $\theta_1 = \theta_2 = \dfrac{\pi}{2}$ this relation may be used to express a_2 in terms of a_1, b_1, and b_2 by an equation of the form

$$a_2 - a_1 \approx - C_1(b_2 - b_1). \qquad \qquad \cdot \qquad \cdot \quad (2)$$

where C_1 is, to a first approximation, a function of a_1, b_1 only, and is of the order of (b/a).

The above equation was obtained by expansion of the elliptic integrals in series of powers of b^2/a^2. For other values of θ_1 and θ_2 this is not so readily done, and the following approximation was made.

It was found graphically that $\theta_1 - \theta_2$, which is of course zero at $\theta = 0, \dfrac{\pi}{2}$, increases smoothly to a maximum at $\theta = \dfrac{\pi}{2}$, remaining always small for adjacent ellipses. Fitting a function to this curve, the form

$$(\theta_2 - \theta_1) = - \eta \sin \theta \cos \theta. \qquad \qquad \ldots \quad (3)$$

was assumed, where η may be determined from the value of $\theta_2 - \theta_1$ at $\theta = \frac{\pi}{4}$, namely

$$\theta_2 - \theta_1 \approx \left\{ \int_0^{\frac{\pi}{4}} \sqrt{a_1^2 - (a_1^2 - b_1^2)\cos^2\theta}\, d\theta \right.$$

$$\left. - \int_0^{\frac{\pi}{4}} \sqrt{a_2^2 - (a_2^2 - b_2^2)\cos^2\theta}\, d\theta \right\} \frac{\sqrt{2}}{\sqrt{a_1^2 + b_1^2}}$$

$$\approx \left\{ \int_{\frac{\pi}{4}}^{\frac{\pi}{2}} \sqrt{a_2^2 - (a_2^2 - b_2^2\cos^2\theta}\, d\theta \right.$$

$$\left. - \int_{\frac{\pi}{4}}^{\frac{\pi}{2}} \sqrt{a_1^2 - (a_1^2 - b_1^2)\cos^2\theta}\, d\theta \right\} \frac{\sqrt{2}}{\sqrt{a_1^2 + b_1^2}}$$

for convenience in evaluating the integrals (App. II.). This finally gives

$$(\theta_2 - \theta_1) \approx -\frac{2\sqrt{2}}{\sqrt{a^2 + b^2}} \, C_2 \sin\theta \cos\theta (b_2 - b_1) \qquad (4)$$

where C_2 is another function of a_1, b_1.

Equations (2) and (4) are needed to ensure that the same point is considered in its initial and final positions. This is necessary in valuating the strain energy, which is given by

$$U = \iint \tfrac{1}{2} D \left[(\kappa + \kappa')^2 - 2(1 - \sigma)\kappa\kappa' \right] dS \qquad (5)$$

integrated over the middle surface of the shell (see ref. 12, p. 504). Here

$$D = \frac{2}{3} \frac{Eh^3}{1 - \sigma^2}$$

where $2h$ is the thickness of the shell, E is Young's modulus and σ Poisson's ratio, and κ, κ' are increments in the principal curvatures.

In a small displacement from equilibrium the work done by the internal pressure is approximately p (increase in enclosed volume) $= p\delta(\pi R_2 X_2 a_2 b_2)$ and must equal the increase in the strain energy

$$p\delta(\pi R_2 X_2 a_2 b_2) = \delta U . \qquad \ldots \qquad (6)$$

giving b_2 as a function of p, and hence the other unknowns.

In expressing the strain energy as a function of b_2 a number of series approximations are needed. The lines of curvature are the lines $\chi = $ constant and $\theta = $ constant, so the principal curvatures are respectively

$$\frac{\cos \Phi}{r}$$

where $\tan \Phi = -\dfrac{dy}{dx} = \dfrac{b}{a} \cot \theta$, and

$$\frac{b}{a^2(1 - e^2 \cos^2 \theta)^{\frac{3}{2}}}$$

where $e^2 = \dfrac{a^2 - b^2}{a^2}$. The quantities κ and κ' are then the changes in

these quantities on going from the point at θ_1 on the first ellipse to θ_2 on the second,

$$\kappa = \frac{\cos \Phi_2}{r_2} - \frac{\cos \Phi_1}{r_1}$$

$$\kappa' = \frac{b_2}{a_2^2 (1 - e_2^2 \cos^2 \theta_2)^{\frac{3}{2}}} - \frac{b_1}{a_1^2 (1 - e_1^2 \cos^2 \theta_1)^{\frac{3}{2}}}$$

Taking the first terms of series expansions in powers of $\dfrac{a_2 - a_1}{a_1}, \dfrac{b_2 - b_1}{b_1}$

and $\dfrac{\theta_2 - \theta_1}{\theta_1}$, and using (2) and (3) these become

$$\kappa \approx \eta \left(-\frac{R_1 \sin \theta_1 \cos^2 \theta_1}{r_1^2 a^{\frac{1}{2}}} + \frac{e_1^2 \sin^3 \theta_1 \cos^2 \theta_1}{r_1 a^{\frac{1}{2}}} \right)$$

$$+ (b_2 - b_1) \left(-\frac{\sin \theta_1}{b_1 r_1 a^{\frac{1}{2}}} - \frac{b_1 \sin \theta_1 \cos^2 \theta_1}{e_1^2 r_1 a^{\frac{1}{2}}} \left(1 + \frac{b_1 C_1}{a_1} \right) \right)$$

$$\kappa' \approx \eta \cdot \frac{3 b_1 e_1^2 \sin^2 \theta_1 \cos^2 \theta_1}{a_1^2 a^{\frac{1}{2}}} + \frac{(b_2 - b_1)}{a_1^2 a^{\frac{1}{2}}} \left(1 + \frac{2 b_1 C_1}{a_1} \right.$$

$$\left. - \frac{3 b_1^2}{a_1^2 a} (1 + \frac{b_1 C_1}{a_1}) \cos^2 \theta_1 \right)$$

where $a = 1 - e_1^2 \cos^2 \theta_1$. These are to be substituted in (5) using (4) to give an expression of the form

$$U \approx (b_2 - b_1)^2 F$$

which is obtained after tedious expansion in series of elliptic integrals (see App. II.).

Then using (6)

$$\delta(p\,\pi R_2 \chi_2 a_2 b_2) = p\,\pi R_1 \chi_1 (b_2 \delta a_2 + a_2 \delta b_2)$$
$$\approx p\,\pi R_1 \chi_1 (a_1 - C_1 b_1) \delta b_2$$
$$\approx 2(b_2 - b_1) F \delta b_2$$

so that

$$b_2 - b_1 \approx \frac{p\,\pi R_1 \chi_1 (a_1 - C_1 b_1)}{2F}$$

This is the method used, but for various reasons it is not satisfactory. Comparison with experimental results on eight gauges at the Division of Metrology, C.S.I.R., showed that the theoretical treatment gave deflections considerably too small, varying from 2/3 to 1/8 of the experimental. This means that the tube of this theory is stiffer than the actual one, whereas Sunatani's[11] method made the tube too flexible, giving from twice to eleven times the deflection.

Several causes may contribute to this lack of agreement, the main one probably being the initial assumption that the ellipse distorts into another ellipse. In passing to the corresponding point on a neighbouring ellipse the change of curvature κ' is small, and comparable with the change on going to a neighbouring point on the same ellipse. Thus if the final form in fact differs only slightly from an ellipse it may make a considerable difference to the result.

This does not mean that results deduced for an elliptic cross section can not be applied to a tube of a slightly different form ; but in estimating the behaviour for the case of the ellipse the final form should really be deduced, not assumed.

The assumption that the deflection of the end of the tube is largely due to inextensional bending was supported to some extent by the experimental results mentioned above. Values of $\dfrac{b_2 - b_1}{b_1}$ and $\dfrac{\chi_1 - \chi_2}{\chi_2}$ calculated from them are given below for comparison. They show that bending theory may well be used to estimate the order of strains involved.

$\dfrac{b_2 - b_1}{b_1}$	·023	·036	·026	·035	·023	·032	·024	·020
$\dfrac{\chi_1 - \chi_2}{\chi_2}$	·031	·026	·020	·028	·023	·025	·025	·026

4. Conclusions

The Bourdon tube straightens under pressure due to the distortion of its cross section towards a circular shade. For thin-walled tubes the effect is due chiefly to inextensional bending of the shell, so that in certain cases the direct stresses may be neglected.

A simplified theory of this kind has been devised here by assuming the form but not the amount of the distortion. On comparing the calculated deflections with experiment they were found to be too small, ranging from 2/3 to 1/8 of the experimental values. It is possible that the results could be improved by retaining second order terms in the series used, but an exact solution is not obtainable by this method.

For real accuracy the form of the strained tube could not be assumed, but would have to be deduced from thin shell theory. A possible approach would be to neglect the direct stresses and strains in the equations of equilibrium and attempt to solve the problem in terms of the two principal curvatures and bending moments.

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Appendix I

Proof that the bending moment about a point on the axis of a closed curved tube due to internal pressure on its walls is zero for all tubes having an axis of symmetry perpendicular to the plane of bending.
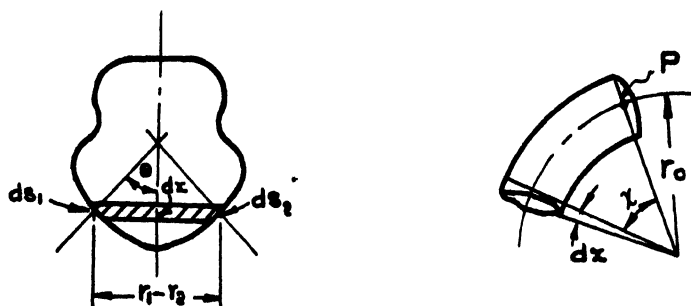
Fig. 3.

Consider the moment about P due to the pressure on an element of the tube which subtends the angle δX at O, a point on the ZZ axis. The same pressure p acts normal to the surface elements ds_1 and ds_2 which are of the same length in the plane of the cross section but differ by $(r_1 - r_2)\delta X$ along the tube. The resultant outward thrust due to pressure is

$$p\,ds\,\sin\,\theta(r_1 - r_2)dX = p\,.\,dA\,.\,dX$$

where dA is the shaded element of area. For the whole cross section it is AdX, with moment about P given by

$$dM = pr_0\,\sin\,XAdX$$

Thus the total moment from elements from $X = 0$ to $X = X_1$ say is

$$M = pr_0A(1 - \cos\,X_1)$$

in a clockwise sense about P.

The moment due to the pressure on the closed end is

$$\int (r - r_0\,\cos\,X_1)pdA'$$

where dA' is now an element of area parallel to the axis and distant r from it. For a symmetrical cross section this reduces to the moment

$$M' = pr_0A(1 - \cos\,X_1)$$

in the anti-clockwise sense.

The total moment about P is then

$$M - M' = 0$$

as required.

Appendix II

In determining η as a function of $(b_2 - b_1)$ it was necessary to express the elliptic integral

$$\int_{\frac{\pi}{4}} \sqrt{a^2\,\sin^2\,\theta + b^2\,\cos^2\,\theta}\,d\theta$$

as a function of a, b. This may be done by expanding as a series in powers of $\dfrac{b^2}{a^2 - b^2}$, converging uniformly if b/a is small.

$$\int_{\frac{\pi}{4}}^{\frac{\pi}{2}} \sqrt{a^2 \sin^2 \theta + b^2 \cos^2 \theta}\, d\theta = \sqrt{a^2 - b^2} \int_{\frac{\pi}{4}}^{\frac{\pi}{2}} \sqrt{1 + \frac{b^2}{a^2 - b^2} \operatorname{cosec}^2 \theta} \,.\sin \theta\, d\theta$$

$$\approx \sqrt{a^2 - b^2} \int_{\frac{\pi}{4}}^{\frac{\pi}{2}} (\sin \theta + \tfrac{1}{2} \frac{b^2}{a^2 - b^2} \operatorname{cosec} \theta$$

$$- \tfrac{1}{8} \frac{b^4}{(a^2 - b^2)^2} \operatorname{cosec}^3 \theta) d\theta$$

$$= \sqrt{a^2 - b^2} \left[\frac{1}{\sqrt{2}} - \tfrac{1}{2} \frac{b^2}{a^2 - b^2} \log_e \tan \frac{\pi}{8} \right.$$

$$\left. + \frac{1}{16} \frac{b^4}{(a^2 - b^2)^2} \left(-\sqrt{2} + \log_e \tan \frac{\pi}{8} \right) \right]$$

The other elliptic integrals which occur in evaluating the strain energy are all complete integrals, that is integrated in the range $0, \frac{\pi}{2}$. They were expanded in powers of b^2/a^2, using the series given by Jahnke and Emde (ref. 13, p. 73). In their notation the integrals

$$K = \int_0^{\frac{\pi}{2}} \frac{d\theta}{\sqrt{1 - e^2 \sin^2 \theta}} = \int_0^{\frac{\pi}{2}} \frac{d\theta}{\sqrt{1 - e^2 \cos^2 \theta}}$$

and

$$C = \int_0^{\frac{\pi}{2}} \frac{\sin^2 \theta \cos^2 \theta\, d\theta}{(1 - e^2 \sin^2 \theta)^{3/2}} = \int_0^{\frac{\pi}{2}} \frac{\sin^2 \theta \cos^2 \theta\, d\theta}{(1 - e^2 \cos^2 \theta)^{3/2}}$$

are given as series in powers of $k'^2 = \frac{b^2}{a^2}$, the coefficients involving $\nu = \log_e \frac{4a}{b}$. Since these series converge uniformly for small values of b/a it is possible to obtain other series expansions by differentiating term by term and forming linear combinations, using the relation

$$e^2 = 1 - \frac{b^2}{a^2}$$

For instance

$$\int_0^{\frac{\pi}{2}} \frac{\sin^2 \theta \cos^4 \theta\, d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}} = \frac{2}{3} \frac{dC}{de^2}$$

$$= -\frac{2}{3} \frac{d}{d(b^2/a^2)} \left(\nu - 2 + \frac{9}{4} (\nu - \frac{5}{3}) \frac{b^2}{a^2} + \dots \right)$$

$$= \frac{a^2}{3b^2} - \frac{3\nu}{2} + \frac{13}{4} + \dots$$

The integral

$$\int_0^{\frac{\pi}{2}} \frac{d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}}$$

is obtained by differentiation and addition

$$\int_0^{\frac{\pi}{2}} \frac{d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}} = K + 2e^2 \frac{dK}{de^2} + \frac{2e^2}{3} \frac{d}{de^2}\left[K + 2e^2 \frac{dK}{de^2}\right]$$

$$= \frac{2}{3} \frac{a^4}{b^4} + \tfrac{1}{2} \frac{a^2}{b^2} + \frac{3\nu}{8} - \frac{7}{32}$$

From this the integrals $\displaystyle\int_0^{\frac{\pi}{2}} \frac{\cos^2 \theta \, d\theta}{(1 - e^2 \cos^2 \theta)^{7/2}}$

and $\displaystyle\int_0^{\frac{\pi}{2}} \frac{\cos^4 \theta \, d\theta}{(1 - e^2 \cos^2 \theta)^{9/2}}$

are readily obtained by differentiation as above.

Integrals containing higher powers of sin θ in the numerator may be obtained by using the formula :

$$\frac{1}{(1 - e^2 \cos^2 \theta)^n} = \frac{1 - e^2}{(1 - e^2 \cos^2 \theta)^{n+1}} + \frac{e^2 \sin^2 \theta}{(1 - e^2 \cos^2 \theta)^{n+1}}$$

for instance :

$$\int_0^{\frac{\pi}{2}} \frac{\sin^4 \theta \, d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}} = -\frac{1 - e^2}{e^2}\int_0^{\frac{\pi}{2}} \frac{\sin^2 \theta \, d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}}$$

$$+ \frac{1}{e^2}\int_0^{\frac{\pi}{2}} \frac{\sin^2 \theta \, d\theta}{(1 - e^2 \cos^2 \theta)^{3/2}}$$

and the integrals on the right are similarly expressed in terms of

$$\int_0^{\frac{\pi}{2}} \frac{d\theta}{(1 - e^2 \cos^2 \theta)^{5/2}}, \int_0^{\frac{\pi}{2}} \frac{d\theta}{(1 - e^2 \cos^2 \theta)^{3/2}} \text{ and } \int_0^{\frac{\pi}{2}} \frac{d\theta}{(1 - e^2 \cos^2 \theta)^{\frac{1}{2}}} .$$

In this manner all the elliptic integrals required are represented approximately by the first few terms of series in powers of b^2/a^2.

A Report on Comi..ercial Tuna Trolling Tests in South-Eastern Australia.

By D. L. Serventy, B.Sc., Ph.D.*

Summary.

A commercial tuna trolling test in the waters of Tasmania and New South Wales in 1941 was sponsored by the C.S.I.R., but the results proved disappointing.

The fishing results are analysed and among the conclusions drawn is the fact that southern bluefin tuna fluctuate strongly in abundance and other characteristics in south-eastern Australia.

1. Introduction.

The Division of Fisheries investigation vessel, M.V. *Warreen,* conducted surveys in south-eastern Australia for tuna and other pelagic fish from the time of its commissioning in May, 1938, to June, 1942, when its operations ceased owing to the war. Attention was directed to three principal methods of fishing for tuna: (1) by trolling; (2) pole-fishing with live bait; (3) purse-seining.

During the period, only a limited amount of tuna had been caught for commercial purposes in Australia, and that almost entirely by trolling. The desirability of proving the possibilities of the troll fishery by a comprehensive commercial test, in association with the C.S.I.R., early became apparent and was made more urgent by war-time needs for processed fish.

The tuna trolling tests were eventually carried out in 1941, in Tasmania and New South Wales. They developed out of a recommendation by Mr. S. Fowler to Dr. H. Thompson (Chief of the Division of Fisheries) that, since he believed the existing research vessel, M.V. *Warreen,* could not give detailed attention to several fisheries over a wide area at the same time, two smaller boats be built for intensive research and demonstration of fishing methods in south-eastern Australia. At the time of this recommendation, it was proposed to build a new research vessel for explorations in Western Australia, and Mr. Fowler recommended that instead of building this vessel the *Warreen* be assigned to Western Australia and that the two smaller vessels take her place in south-eastern Australia to fish intensively in restricted areas for tuna and other species. They would demonstrate whether or not tuna could be trolled in commercial quantities with reasonable consistency over a period. If these craft were not ready in time, he advocated that a Tasmanian tuna fishing boat, the *Weerutta,* be chartered to fish intensively in the Flinders Island and north-east Tasmanian areas from about February to July following (namely, in 1941).

The proposed small research vessels were not built, and Dr. Thompson recommended to the Chief Executive Officer that the *Weerutta* be chartered for nine months for tuna and barracouta

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*   *An officer of the Division of Fisheries.

fishing in New South Wales and Tasmania. Approval of the tests was not obtained to take advantage of the current tuna season in *New South Wales*, but arrangements were completed for the carrying out of the test with two vessels in Tasmania from March to June, 1941. The tests were later extended to the New South Wales coast during mid-August to the end of November, 1941.

The tests themselves were organized by Mr. Fowler, and in place of outright chartering the two vessels concerned were subsidized by the C.S.I.R. In general, the agreement with the owner-skippers provided for the guaranteeing of each vessel for a £500 catch during a four months' fishing period, to cover the main tuna season in particular areas. The amount of the guarantee was stipulated by each owner as a condition of his agreeing to engage in the tests, and was based on his estimate of the costs of operation, plus reasonable remuneration. The owners bore all risks in respect of vessels, gear, and personnel. The contract set out that " the primary object of the test is the capture of tuna and barracouta with most importance attaching to tuna. If, however, conditions at any time are not suitable for the capture of these species, such opportunities as might be presented for the capture of other marketable species shall be seized, and the value of such catches shall be included in the total catch for which the guarantee is offered." It was arranged that the M.V. *Warreen* should continuously patrol the fishing area during the period of the tests for the information of the vessels participating, and that any fish caught by her should be disposed of to make up any payments that might have to be made under the terms of the guarantee.

The vessels selected for the test were two 55-ft. Tasmanian power ketches, the *Weerutta* and the *Jean Nichols*. The *Jean Nichols* was a new vessel, but the *Weerutta* had been converted for tuna trolling in 1939, and had fished during the Tasmanian seasons of 1939 and 1940.

Two separate tests were arranged. The first was in the east Tasmanian area, from Flinders Island to Cape Pillar, the *Weerutta* fishing from March to June and the *Jean Nichols* from mid-March to mid-July. The second test was made on the south coast of New South Wales from August to November inclusive, in which only the *Weerutta* participated. Both vessels used the same gear, of the pattern employed on the *Warreen* (C.S.I.R. Pamphlet No. 104), and trolled as a rule eight lines, at a speed of 8 knots. Bone jigs were mainly used for tuna fishing, but recourse was also had to wood jigs and feather lures.

General Results.

In both of the tests the vessels failed to reach their quotas under the guarantee, and in each instance ended the season with considerable deficiencies. In the Tasmanian test the *Weerutta* and the *Jean Nichols,* after allowing for the fish caught by ,the *Warreen,* showed a total deficiency of £380 8s. 9d. on the total guarantee of £1,000, and in the New South Wales test the loss was £383 0s. 6d. on a guarantee of £575 14s. 3d. Thus, the financial cost to the C.S.I.R. of the combined experiments was £763 9s. 3d. The detailed account of the fishing operations and assessment of the results will be described for the two areas separately.

2. The Tasmanian Test.

i. General.

The *Weerutta* operated mostly in the south-east Tasmanian region, principally in the vicinity of Cape Pillar, whilst the *Jean Nichols* was mostly fishing in the Furneaux Island region. Their respective performances were as follows:—

	Weerutta.		*Jean Nichols.*		*Warreen.*	
	Days.	Per cent.	Days.	Per cent.	Days.	Per cent.
Actual fishing days	69	57	67	56	76	80
Weatherbound ..	10	8	14	12	8	8
In port	37	30	26	22	11	12
Under repairs .	6	5	12	10	—	—
	122 = 17·4 weeks.		119 = 17 weeks.		95 = 13·6 weeks.	

The *Weerutta* tried for tuna on 63 of the 69 fishing days, trolling for an aggregate of 410 hours, or 6·5 hours per day; the balance of the time was devoted to other types of fishing. The *Jean Nichols* tried for tuna on 47 days, and trolled for an aggregate of about 474 hours, or 10 hours per day; the balance of the fishing time was devoted to barracouta fishing. The *Warreen* tried for tuna on 71 days, trolling for an aggregate of 524 hours, or 7·4 hours per day.

In the following table of fish caught, the amounts transferred to the vessels at sea by the *Warreen* and included above under " General Results " have been deducted:—

Kind of Fish.	*Weerutta.*				*Jean Nichols.*		
	£	s.	d.		£	s.	d.
Tuna	124	7	6	..	80	5	5
Barracouta	90	16	0	..	126	8	3
Horse mackerel ..	33	14	9	..	—		
Salmon	10	14	5	..	—		
Various	19	1	2	..	—		
	278	13	10	..	206	13	8

ii. Economics.

The following analysis has been prepared of the working expenses and earnings of the two boats during the test:—

Fishing Boat *Weerutta.*

	£	s.	d.		From All Fish. £ s. d.	From Tuna Only. £ s. d.
Total earnings ..	..			..	278 13 10	124 7 6
Expenditure—						
Fuel oil ..	45	0	7			
Lubricating oil	4	16	0			
Ice	28	4	3			
New fishing gear	10	12	6			
Total expenditure ..	..			..	88 13 4	88 13 4
Net return for boat ..	..			..	190 0 6	35 14 2
Return for boat per week ..	..				10 18 5	—
Return for boat per week if tuna only had been fished for ..	..				—	2 1 0

The approximate value of the fishing gear on the boat at the start of the test was £32. The crew comprised three men. Depreciation, insurance, and wages would be additional charges on boat earnings.

Fishing Boat *Jean Nichols.*

	£	s.	d.	From All Fish. £ s. d.	From Tuna Only. £ s. d.
Total earnings			..	206 13 8	80 5 5
Expenditure—					
Fuel oil ..	75	18	8		
Lubricating oil	1	6	8		
Ice	8	0	0		
Fishing gear ..	10	0	0		
Total expenditure			..	95 5 4	95 5 4
Net return for boat ..			..	111 8 4	—
Return for boat per week ..			..	6 11 1	—
Net loss during period if tuna alone had been fished for				—	15 0 0

iii. The Tuna Catches.

Tuna were the primary objective of the tests, and most attention was paid to fishing for these species. The other fish taken were sought only incidentally, and when there were no tuna at the time. The following table is an analysis of the tuna fishing performances of the two boats compared with that of the *Warreen,* which was fishing in the same area for an equivalent period*:—

Species.	Weerutta. £ s. d.	Jean Nichols. £ s. d.	Warreen. £ s. d.
Bluefin ..	123 2 0	56 7 6	80 2 0
Striped tuna ..	0 14 6	26 8 0	59 4 6
Albacore ..	0 11 0	2 0 6	1 11 0
	124 7 6	84 16 0	140 17 6

* The slight differences between the values in this section and that in the preceding paragraphs are due to the fact that the data which follow are computed from the fishing boats' logs and the *Warreen* record sheets. Often numbers of fish only have been recorded by the boats, and the computed weights differ slightly from the cannery returns.

In quantities of fish, the details are as follows:—

Species.	Weerutta.		Jean Nichols.		Warreen.	
	No.	Weight.	No.	Weight.	No.	Weight.
		Tons.		Tons.		Tons.
Bluefin ..	603	4·924	251	2·215	363	3·204
Striped Tuna	9	0·029	325	1·056	729	2·369
Albacore ..	8	0·022	29	0·081	22	0·062
		4·975		3·352		5·635

To meet the guarantee of £500 per boat would have required a weekly average catch of about 1¼ ton. In actual fact, the catch of the *Weerutta* represented a weekly average of 0·29 ton, and that of the *Jean Nichols* of 0·20 ton. In the former case it was only 23 per cent. of the required quota, and in the latter only 16 per cent. The *Weerutta* had been fishing for tuna for six weeks before the tests officially started, but the amount caught during that period would not have affected the position materially. For the entire season the weekly average catch (for bluefin) was only 0·27 ton.

The difference in the catches of bluefin and striped tuna are due to the fact that striped tuna were confined to the Flinders Island and north-east Tasmanian region, where the *Jean Nichols* was mainly fishing, while the bluefin were more abundant in south-east Tasmania, where the *Weerutta* operated. The *Warreen* worked in both areas. The higher catch of the *Warreen* in striped tuna over that of the *Jean Nichols* may be partly accounted for in that the latter vessel began fishing just after the peak of the striped tuna concentration.

The frequency with which the various daily catches of bluefin were made by the *Weerutta* is given on p. 146.

iv. Interpretation of the Tests.

(i) *Southern Bluefin Tuna* (Thunnus maccoyii).

The value of the tests in assessing the tuna trolling possibilities of the Tasmanian region may be set in a better perspective by a consideration of other data possessed by the Division. This has been gained from two sources: (1) the sampling of the *Warreen*, and (2) the commercial fishing of the *Weerutta* during the 1939 and 1940 and part of the 1942 seasons.

(a) *The " Warreen's " sampling.*—Since May, 1938, the *Warreen* has trolled at frequent intervals in the south-east Australian littoral region, and data have been accumulated since October of that year on a basis of catch per unit of gear. A general review of her trolling operations is now in course of preparation and, though it is not possible to give here a summary of the quantitative results as in the

case of the New South Wales coast (see p. 148), it may be stated definitely that, during the period of the surveys and over the areas covered, the *Warreen* had recorded generally higher catch per unit of gear figures in New South Wales waters than in Tasmanian. Further, in the Tasmanian region itself, the figures are higher for south-east Tasmania, around Cape Pillar, than they are for the Flinders Island region.

(b) *The commercial fishing results.*—For this information we have as the only extant source the tuna trolling ventures of the ketch *Weerutta,* carried on since 1939 in south-east Tasmania.

In 1939, the *Weerutta* started fishing for tuna on May 9 and discontinued on August 4. During these three months, the boat caught 6·7 tons of tuna, which at £25 per ton was valued at £168. In 1940, fishing was begun on February 26 and ended on August 2, when the tuna took off. In this period, of just over five months, the boat caught 7·37 tons of tuna, valued at £184. In 1942, the year following the tests, the *Weerutta* endeavoured to resume tuna fishing at the start of the season, but the results were so disappointing that the venture was abandoned and her activities were diverted to other types of fishing. The tuna catch in all these years was almost entirely of southern bluefin.

It is rather difficult to reduce the preceding catch figures to a uniform basis for direct comparison. The varying length of the season with a varying abundance of fish precludes the use of a direct *pro rata* comparison. It was decided, therefore, to take a four months' period, covering the main part of the season, as a base. The figures for 1940 were reduced by eliminating the first and last half-months of the catches, whilst for 1939 an approximation of a four months' catch was arrived at by building up the earlier part of the season by following the trend of the catch curve. It was considered also that a fairer assessment of the real position in 1941 would be to take the four months' period as covering the months February to May, instead of the official test period of March to July.

The amended figures thus arrived at for a four months' season are as follows:—

	1939.	1940.	1941.
Quantity caught ..	7·9 tons.	6·7 tons.	6·3 tons.
Value	£198.	£168.	£158.

The three years' catches, therefore, do not vary very widely, and, despite the cumulative experience of the *Weerutta* and the improvement of her working conditions, a decline is shown in catches as compared with 1939. This fall is partly explained by the biological work of the *Warreen* in these waters and the study of the *Weerutta's* own catches during the period.

This is not the place to give a detailed analysis of these data, which will be reported in full in due course. A brief summary will suffice, however, to indicate the nature of the fluctuations which have been met with during the period of study.
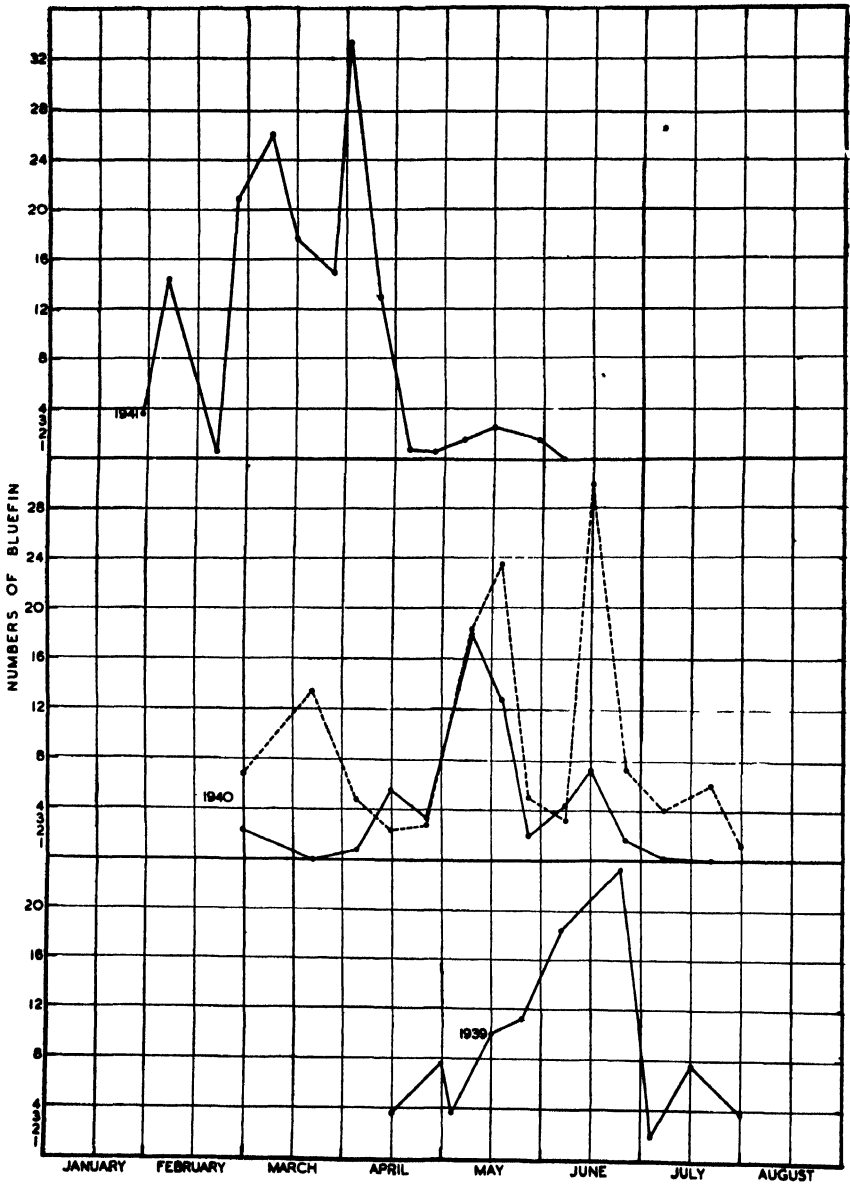
FIG. 1.—Operations of the ketch *Weerutta*.

Catch per day's absence—

————— three-year-old fish,

---------- two-year-old fish.

Fig. 1 gives an analysis of the catches of the *Weerutta* for each of the three seasons on a basis of catch per unit of effort, with the fish caught divided into size (age) groups. This shows the quite striking differences in the duration of the seasons as well as the differences in size or age composition. The season 1940 differs from 1939 and 1941 in that two groups of tuna were present, whereas only one (the larger) occurred in the last-mentioned years. The group on which the fishery fundamentally depends is the three-year-old group during the latter part of its growing season; in 1940 it was reinforced by the two-year-old group.* It will be seen also that the peak of the fishing season has progressively advanced during the three years, and in 1939 the whole of the catch was made in the months *after* the fish had taken off in 1941. As has previously been stated, the *Weerutta* was fishing from January 21, six weeks prior to the start of the official test. Her whole catch during the entire fishing period totalled 811 bluefin, the following being the monthly summaries:—

Month.		Monthly Totals.		Cumulative Totals.	
			Per cent.		Per cent.
January	..	40 Bluefin	4·9	40 Bluefin	4·9
February	..	167 Bluefin	20·6	207 Bluefin	25·5
March	..	382 Bluefin	47·1	589 Bluefin	72·6
April	..	185 Bluefin	22·8	774 Bluefin	95·4
May	..	36 Bluefin	4·4	810 Bluefin	99·8
June	..	1 Bluefin	0·1	811 Bluefin	99·9

Thus it will be seen that almost three-quarters of the fish were taken before the end of March, and practically the whole by the end of April. However, even if the tests had begun on January 21 the tuna results would not have been materially different. The weekly average for the four months' period beginning on that date is only 0·36 ton or 29 per cent of the 1·25 tons which had been reckoned on. The weekly average for the entire season during which fishing had been conducted, namely, 23·4 weeks, was only 0·27 ton.

An important cause of the weight variations in the total catch is not brought out in Fig. 1. This concerns the differences in the average weight of the size-groups from year to year. One of the striking features of the tuna work in 1940 was the demonstration that the two-year-old fish had suffered a serious retardation in growth rate during the growing period of 1939-40, though they entered the fishery at the usual size of this group. This meant that instead of the three-year-old fish averaging about 25½ lb. per fish during the autumn fishery in Tasmania (i.e., at the end of their growing season), as they did in 1939, the fish caught by the *Weerutta* in 1941 averaged only 18¼ lb. Had the three-year-old fish weighed as much as they did in 1939, the *Weerutta's* catch of bluefin during the test would have weighed 6·9 tons instead of 4·9 tons, and the total earnings of the boat would have been £173 instead of the £123 she actually did earn from bluefin tuna by her own fishing efforts. The year 1940 was inferior to 1939 because the larger three-year-old group was not so abundant. thus depressing the average weight of individual fish caught.

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* See Serventy (1941).—The Australian Tunas. Coun. Sci. Ind. Res. (Aust.), Pamph. No. 104, p. 29, for a summary of the growth rates in the southern bluefin and the movement of the various age groups.

In actual numbers of fish landed by the *Weerutta,* the following are details of the three seasons under discussion (the total for 1941 is given, not merely for the test period):—

Year.	Season.	Two-year-old.	Three-year-old.	Total.	Av. Wt. per Fish
1939	3 months	0	587	587	25½ lb.
1940	5 „	710	308	1,018	16¼ lb.
1941	5 „	0	811	811	18¼ lb.

On a four months' basis, reducing the figures in the manner previously stated, the numbers of three-year-old bluefin caught by the *Weerutta* are as follows:—

1939	..	.. 689 individuals.
1940	..	.. 294 individuals.
1941	..	.. 770 individuals.

(c) *Conclusion.*—The available evidence shows that trolling for tuna in Tasmania by present methods is not a commercial proposition taken on its own, but it may be important as an adjunct to other types of fishing, particularly in " good " tuna years. Whether the present low catches are capable of increase by changes in the technique of trolling, greater experience, &c., of the fishermen are matters for the future. It is evident, however, and this aspect is further elaborated later in the New South Wales section of this report, that sharp fluctuations occur in the character of the bluefin stocks, and these must be seriously reckoned with in any local fishery. Some of the factors may cancel each other out in any one year, but their existence is a warning that the returns for the same amount of effort may vary sharply in different years.

(ii) *Striped Tuna* (Katsuwonus pelamis).

The season under review was a good one for striped tuna, according to the experience of the *Warreen*. Very extensive shoals were met with over long periods. Because on an average only about one fish in ten which was hooked by the vessel was actually landed (owing to the weakness of the jaws) trolling is too wasteful, at least when employing the present methods and technique, to warrant a continuance of commercial trolling for this species. There are, however, decided prospects for live-bait fishing.

(iii) *Albacore* (Thunnus germo).

All the experience of the Division suggests that this is a comparatively scarce species in inshore waters where present-day trolling operations are conducted. In 1938 they appeared to be more plentiful than in subsequent years. However, it may be ruled out of consideration for trolling ventures as now conducted.

3. The New South Wales Test.

i. General.

The extension of the guaranteed commercial tests to New South Wales was done on the recommendation of the Fisheries Advisory Committee of the C.S.I.R. at its meeting on June 12, 1941, and concurred in by Mr. Fowler. One of the Tasmanian boats, the *Weerutta,* was engaged on similar terms to those of the preceding

test. The period chosen from mid-August to the end of. November, 1941, covered the main part of the southern bluefin season in New South Wales. A summary of the vessel's performance is as follows:—

	Days.	Per cent.
Actual fishing days	63	67
Weatherbound	21	22
In port	10	11
	94 days = 13·4 weeks.	

All these 63 fishing days were devoted to tuna fishing (there were exactly the same number of tuna-fishing days in the Tasmanian test).

The fish caught were as follows:—

Kind of Fish.	Number.	Weight (in tons).
Southern bluefin ..	1,042 ..	7·845
Striped tuna ..	2 ..	0·004
Albacore	18 ..	0·095
Bonito	193 ..	0·462
Total weight of tuna	..	8·406
Kingfish (*Seriola*) ..	25 ..	0·124
Total weight of trolled fish	..	8·530

The total weight credited to the vessel at the cannery was 7·583 tons of tuna and 0·124 ton of kingfish. All were paid for on the basis of £25 per ton, namely, a total of £192 13s. 9d. The discrepancy of 0·823 ton is due partly to the loss of some bluefin whilst crossing the bar at Narooma, and the rejection by the cannery of approximately 928 lb. of bonito. In the calculations concerning tuna productivity, the economics of which follow, cognizance is taken of the whole original catch and not of the net cannery figures.

ii. Economics.

The following is an analysis of working expenses and earnings during the test:—

	£	s.	d.
Gross earnings (from tuna only)	210	3	0

Expenditure—	£	s.	d.			
Fuel oil	49	14	7			
Lubricating oil ..	7	15	0			
Ice (12 tons 8 cwt.) ..	36	17	9			
New fishing gear ..	7	12	6			
Total expenditure				101	19	10

	£	s.	d.
Net return for boat ·(for 13·4 weeks) ..	108	3	2
Return to boat per week	8	1	5

iil. The Tuna Catches.

The catch of southern bluefin during the period averaged 0·585 ton per week, compared with 0·29 ton by the same vessel in the earlier Tasmanian test. The average catch per fishing day was 278·9 lb. in N.S.W. and 177·9 lb. in Tasmania, a ratio of 1·6 : 1. For total tuna, the average weekly catch in N.S.W. was 0·627 ton. Bluefin, as has been pointed out, was virtually the only species caught in Tasmania by the *Weerutta*.

The following table shows the frequency with which various daily catches were made by the *Weerutta*, both in New South Wales and Tasmanian tests:—

No. of Bluefin Caught per Day.	Number of Days. N.S.W.	Tasmania.
No fish	19	26
1– 10	26	18
11– 20	5	8
21– 30	3	5
31– 40	0	3
41– 50	3	1
51– 60	0	0
61– 70	1	2
71– 80	3	0
81– 90	0	0
91–100	1	0
101–110	0	0
111–120	0	0
121–130	1	0
131 -140	0	0
141–150	1	0
	63	63

iv. Interpretation of the Test.

Though no commercial trolling of any moment similar to that of the *Weerutta* in Tasmania has been conducted in New South Wales, the surveys of the *Warreen* lend themselves better to analysis than those further south. The significance of the test and its value in assessing the productivity of bluefin in New South Wales can be discussed on somewhat different lines, therefore, from those employed in the Tasmanian tests.

The following paragraphs, extracted from the report of the 22nd cruise of the *Warreen*, give an indication of the characteristics of the season:—" According to the catch per unit of gear figures of the *Warreen*, the present season (1941) is a very poor one, and is only about half as good as the peak season of 1939. The predominant age-group, the third-year fish, averaged about 17 lb. (i.e., at the beginning of the growing season) and was thus normal in size, compared with last year. Striking features of this year's age-composition of the bluefin stocks were the dearth of large four-year-old fish, and also of the small two-year-old class. This was the case last year as well. Another noteworthy feature was that this year the

bluefin did not penetrate much further north than the Montague Island region, whereas in 1939 they reached Sydney. For the first time since the Division's investigations began there was no tuna fishing at Jervis bay."

The use of the standard trolling gear by the *Warreen* on the New South Wales coast during 1938, 1939, 1940, and 1941, when it was operated in a fairly consistent manner throughout, enables the computation of index numbers for each season to give a numerical expression of the varying abundance of the bluefin.

As the average weight of the fish caught varies widely each season, for commercial purposes the numerical index must be weighted to take account of this factor.

By themselves, of course, the index numbers obtained by the *Warreen* cannot be directly applied commercially except insofar as they reveal the fluctuation which exists from year to year. In the season of the test, however, by utilizing the data obtained from the operation of the *Weerutta,* the catches of the two vessels could be linked. By interpolation the index numbers of the *Warreen* for the earlier years can then be given a direct commercial interpretation.

In the following table the column " commercial index " refers to the numerical index as weighted to take account of the average weight of the fish for the season. The commercial values are computed from the *Weerutta's* gross earnings for the test season, reckoned at £25 per ton. The year 1939 was selected as the base year, with the index at 100:—

Year.	Numerical Index.	Mean Wt. of All Fish.	Commercial Index.	Av. Expected Catch per Week.	
				Av. Tons.	Av. Value.
1938	45	lb 10·27	29	0 414	£10 7 0
1939	100	16·16	100	1·427	35 13 6
1940	81	12·39	62	0 885	22 2 6
1941	42	15·75	41	0·585	14 12 6

It will be seen that the season 1939 was by far the best of the four which had been investigated, and it may be fairly said that of these four seasons only one, that of 1939, offered a reasonably attractive return to troll fishermen.

The *Warreen* passed out of the control of the C.S.I.R. to the Royal Australian Navy in 1942, and no effective means remained of checking on the tuna stocks. From the experience of fishermen and other evidence, however, it was clear that the 1942 season was a very poor one. The 1943 season showed a pronounced recovery, and southern bluefin was abundant during the last part of the season. Such evidence as was available indicated that the 1944 and 1945 seasons were also better than the test season of 1941.

The results show that, with the methods used, trolling for southern bluefin on the New South Wales coast can only be regarded as a border-line fishery, suited to the operations of small boats with low overhead costs. The matter is entirely one of economics, however, and were the Californian scale of prices ruling a much brighter position would be shown. On Californian standards, the southern bluefin is under-valued locally, and in the following table the computed values of the.New South Wales catch are compared with equivalent Californian figures (at £32 per ton, *vide* " The Australian Tunas," C.S.I.R. Pamphlet 104, p. 11):—

Year.	Average Return per Week (Bluefin only).	
	@ Ruling N.S.W. Rates.	@ Californian Rates.
1938	£10 7 0	£13 4 11
1939	35 13 6	45 13 3
1940	22 2 6	28 6 5
1941	14 12 6	18 14 5
Average Weekly return for the season (Aug.-Nov.)	20 13 11	26 9 9

As an approximate check on the validity of the method of assessment, it is of interest to compare the tuna production figures for the Merimbula district kept by Mr. W. L. Packenham, the local fishery inspector, since 1937. Unfortunately, no similar data are available for other districts.

The amount of bluefin caught is as follows:—

	Year—				
	1937.	1938.	1939.	1940.	1941.
Boxes ..	148	4	542	38	125

Prior to 1940 only the Melbourne bait market was available to these fishermen. In 1940 the Eden cannery came into the market for tuna, and could be regarded as providing an added stimulus to tuna fishing. In 1941 the Narooma cannery entered into general production and was prepared to take all the tuna offering. The figures clearly show that 1939 was an · outstanding year, but they probably exaggerate its superiority over other years, as in the good year when fish are plentiful everybody is out after them. In poor years the tuna are mostly left alone and effort is concentrated on other species.

Some further remarks may be made on the index figures. The years 1938 and 1941 were incompletely sampled as compared with the other seasons, but are comparable with each other. The low average weight of fish in 1938 is due to the predominance in that year of the smallest size-group of tuna, though it is likely the average would have

been raised slightly had the earlier part of the season been better sampled. In 1941 there was a noteworthy scarcity of these small fish. The index numbers themselves should be regarded as approximate; they were prepared on a system which took account of the shrinking of the tuna area as the season advanced, and this required some arbitrary adjustment of the figures. However, each year was treated in a comparable manner and, therefore, their relative values may be accepted as approximating reasonably near to the real situation. Fuller details will be given when the trolling results of the *Warreen* during her surveys in south-eastern Australia are published.

Appendix.

Weather Conditions—Tasmanian Area.

The Director of Meteorological Services (Group-Captain H. N. Warren) has provided the following data on the weather conditions in the area under test:—

" It has not been possible to arrive at more than a very general estimate of how wind and sea conditions varied from normal in the period March 1 to June 30, 1941. Statistical records of wind and sea conditions have not been tabulated in the past.

From an inspection of mean and normal pressure gradient maps, which show differences in air pressure between various stations on which wind force depends, the following notes have been prepared, which give a general indication of the characteristics of these months:—

March: *Flinders Island Area.*—The pressure gradient indicated winds above average strength or frequency from a westerly direction. A period of exceptionally low pressure was experienced from March 20–28.

East Coast of Tasmania.—Ditto—but winds more from south-west or south than normally.

April: *Flinders Island Area.*—Winds rather above the average, easterly tendency, but on the whole a quieter month than normally.

East Coast of Tasmania.—Average or slightly over average winds, with a south-west or southerly tendency.

May: *Flinders Island Area.*—The gradient indicated rather higher wind strength than normal from west or south-west, or a higher frequency of winds from this direction.

East Coast of Tasmania.—Ditto—though Tasman Island report showed a prevalence of north-westerlies, strength would be considerably higher than normal.

June: *Flinders Island Area.*—Somewhat stronger winds than normal from west. •

East Coast of Tasmania.—Under average wind strength from west or south-west.

The state of sea would naturally correspond to strength of the wind and whether the latter were onshore or offshore. The normal gradient is for west or south-west winds over this area."

Summary.—The general trend indicated by the pressure charts during the period covered by the inquiry could be stated as somewhat rougher conditions than normal, but not markedly abnormal.

WEATHER CONDITIONS—NEW SOUTH WALES AREA.

The Director of Meteorological Services has supplied the following data on weather conditions in the area under test:—

" I regret that very little information is available concerning wind and sea conditions compared with normal between Tuross Inlet and Cape Howe during the period August to November inclusive, 1941.

We have an anemometer at Moruya Heads, and mean daily wind speeds for each of the four months were as follows:—

August—7·8 m.p.h. (0·7 m.p.h. below the average).

September—9·2 m.p.h. (0·2 m.p.h. above the average).

October—9·8 m.p.h. (0·7 m.p.h. above the average).

November—8·2 m.p.h. (22 days) (0·9 m.p.h. below the average).

Wind directions at Moruya were:—

August.—Mainly S. to S.W., strong on 2 days.

September.—Mainly S.W. in the morning, N.W. or N.E. in the afternoon, strong on 4 days.

October.—Mainly S.E. to S.W. in the morning and E. to S. in the afternoon, but a fair percentage of N.E., strong on 5 days.

November.—Mainly N.E. to S.E., strong on 3 days.

At Gabo Island winds were as follows:—

August.—Majority W. to S.W., strong to gales on 9 days.

September.—Majority N.W. to S.W., strong on 4 days.

October.—Mainly W. to S.W., strong or gales on 15 days.

November.—Mainly W. to S.W., strong on 11 days.

On the whole the season appears to have been about an average one. Mean pressure was above the average in August and November, and below the average in September and October."

NOTES

The Utilization of German Technologists and Scientists in Australia

The Minister for Post-War Reconstruction (Hon. John J. Dedman) has announced that a scheme, appertaining to reparations, has been approved by Federal Cabinet, which should be of interest to Australian industrialists and research institutions. Under this scheme it is proposed to bring into Australia, under certain safeguards, scientists and technologists from Germany. The Australian scheme will be similar to the plans adopted by the United Kingdom, U.S.A., and France, which are already operating for the acquisition of further knowledge of the latest scientific developments that have taken place in Germany.

Technical experts who are required, and who volunteer to come to Australia, will, if politically acceptable, enter Australia for a definite period under contract to the Commonwealth Government. They will be subject to supervision and will have the normal social service and taxation responsibilities of residents. In all cases, these skilled men will be employed only if no Australian, or United Kingdom citizen who is available to migrate here, has the necessary skill and knowledge for that particular sphere of endeavour.

With the introduction of these proposals it is hoped that new industries may be developed here and that existing ones will benefit appreciably from the overseas experience that will be made available to them. For example, in some branches of pure research, metallurgy, electronics, utilization of brown coal, the chemical industry, the production of aluminium, German knowledge far outstrips our own, and Australian industry is now in the position to acquire valuable working information from outstanding German specialists. The services of specialists brought out under Commonwealth Government sponsorship will be available to State authorities and interested private firms who apply for these men.

A Committee which has been set up will consider the requirements of Australia, the qualifications of all German specialists for whom application is made or who may apply, and will advise the Secondary Industries Division of the Department of Post-War Reconstruction—which will be the administrative authority in Australia—as to the suitability of suggested scientists and technologists. This Committee is broadly constituted, having a representative each from the Associated Chamber of Manufactures, Combined Australian Universities, the Commonwealth Office of Education, the Council for Scientific and Industrial Research, the Australian Council of Trade Unions, the Department of Immigration, and the Secondary Industries Division.

The Secondary Industries Division will welcome any inquiries from Australian firms for suitable personnel where it is believed that the introduction of such men could make a contribution to the development of Australian secondary industries. The Commonwealth Government is anxious to make such specialized knowledge available to those who can best use it and so derive the maximum benefit for Australia.

Catalogue of Scientific and Technical Periodicals, New Edition

The following list is supplementary to those published in previous issues. Libraries possessing items with which they are not credited are requested to forward missing entries showing holdings, to Mr. E. R. Pitt, care of C.S.I.R. Information Service, 425 St. Kilda-road, Melbourne, S.C.2, for inclusion in the Catalogue. As full information as possible is given, and bibliographical accuracy cannot be guaranteed. Later particulars are given of a few periodicals included in former lists.

NEW SCIENTIFIC AND TECHNICAL PERIODICALS.

Aero review. Rochester, *N.Y.* V.1, No. 1 = Feb., 1946. *Monthly.* 25c., $3·00.

Agricultural chemicals. New York. *Monthly.*

American aquarist. V.1, No. 1 = Feb., 1946. *Monthly.* 25c., $2·50.

American journal of medicine. New York. V.1, No. 1 = July, 1946. *Monthly.* $10·00.

American medical women's association. Journal. Nashville. V.1, No. 1 = Apr., 1946. *Monthly.* 35c., $3·00.

Atom. Denver, *Col.* V.1, No. 1 = Fall, 1945. *Quarterly.* 25c., $1·00.

Atomic age. Sea Cliff, *N.Y.* V.1, No. 1 = Jan., 1946. $1·00. [Changed to $2·00 with V.1, No. 3, Mar., 1946.]

Atomic engineering. New York. V.1, No. 1 = Sept., 1945. *Irreg.* 25c. per copy (processed).

Atomic information. (National committee on atomic information.) Washington, *D.C. Monthly.*

Atomic scientists of Chicago. Bulletin of the atomic scientists. *Semimonthly.* (N.CSIR:SR; V.CSIR:HO.)

Ceramic association of New York. Monthly news letter. Albany, *New York. Monthly.*

Coal technology. New York. V.1, No. 1 = Feb., 1946. *Bi-monthly.* $1·50, $6·00.

Corrosion and material protection. Pittsburgh, *Pa. Monthly.*

Diesel journal. Jersey City, *N.J.* V.1, No. 1 = Jan., 1946. *Monthly.* 35c., $3·00.

Duodecimal bulletin. (Duodecimal society of America.) Staten Island, *N.Y. Irreg.*

Engineer and foundryman. Johannesburg. *Monthly.*

Fonderie. (Association technique de fonderie.) Paris. *Monthly.*

Food freezing. New York. V.1, No. 1 = Nov., 1945. *Monthly.* 35c., $4·00.

Frozen food industry and locker plant journal. New York. V.1, No. 1 = Nov., 1945. *Monthly.* 50c., $3·00.

Frozen food merchandising. New York. V.1, No. 1 = Mar., 1946. *Monthly.* $3·00.

Fur and feather magazine. (National fur and feather association.) Sliema, *Malta. Quarterly.*

Geriatrics. Minneapolis, *Minn.* V.1, No. 1 = Jan.-Feb., 1946. *Bi-monthly.* 50c., $3·00.

Industrial diamond review. London. *Monthly.*

Journal of the history of medicine and allied sciences. New York. V.1, No. 1 = Jan., 1946. *Quarterly.* $2·50, $7·50. (N.SPH.)

Lubrication engineering. Chicago. V.1, No. 1 = June, 1945. *Quarterly.* 75c., $3·00.

Mining and metallurgical society of America. News letter.

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Reinforced concrete review. (Reinforced concrete association.) London. *Semi-annual.*

Science illustrated. New York. V.1, No. 1 = Apr., 1946. *Monthly.* $3·00.

Sight. Pittsburgh, *Pa.* V.1, No. 1 = Apr., 1946. *Monthly.* 10c., $1·00.

Southern refrigeration journal. Memphis, *Tenn. Monthly.*

Telectronic news. (Television film industries corp) New York. *Monthly.*

Television showman. New York. *Bi-weekly.*

Reviews

" THE USE OF HETEROSIS IN THE PRODUCTION OF AGRICULTURAL AND HORTICULTURAL CROPS," by T. Ashton.

(Imperial Bureau of Plant Breeding and Genetics, 1946, pp. 30 Price 3s. (stg.). Obtainable from the Imperial Agricultural Bureaux, Agricultural Research Building, Penglais, Aberystwyth, Wales.)

This bulletin brings together summaries of a large number of papers which have a bearing on the possibilities of commercial exploitation of hybrid vigour in agricultural and horticultural crops. There are four main sections to the bulletin dealing, respectively, with heterosis in self-pollinated, cross-pollinated, and asexually propagated plants and in forest trees. Within these four sections each crop is treated individually. In the case of maize, only papers on certain fundamental problems are included.

There is an increasing indication that the systematic use of heterosis in commercial crop production offers important possibilities in other crops besides maize, where hybrid production is now an established and well-known practice in the United States. Though the cross-pollinated species are the most promising, the self-pollinated also appear to offer definite prospects, especially those in which a large number of seeds is produced from a single flower, hybridization is a relatively easy process, and the plants are widely spaced in cultivation. These include tobacco, tomato, and egg plant. The value of heterosis is more limited in the case of the normally self-fertilized plants in which one flower produces only one seed and artificial hybridization is a difficult operation, as in the case of wheat, oats, barley, sorghum, and rice.

The bulletin concludes with a useful bibliography of 234 references.

W.I.

"HINTS TO PROSPECTORS AND OWNERS OF TREATMENT PLANTS,"
edited by H. A. Corbet.

(Issued by the Royal Mint, Perth, Western Australia, 1946, pp. 80. Price 1s.
Obtainable from Government Printer, Perth, Mining Registrars throughout
W.A., and other sources.)

This publication was brought out by the Royal Mint, Perth, for
the benefit of men in isolated mining ventures. It includes useful
hints on prospecting, dry blowing, miners' rights and regulations,
geology, batteries and cyaniding, preparation of samples for assaying,
use of explosives, first aid and health; reference is also made to
prospecting for other economic minerals. For this reason, while
details relate more specifically to the Western Australian goldfields,
the booklet has a much wider appeal to all those who propose
prospecting or are engaged in the goldmining industry in remote
regions where limited facilities and difficult conditions pertain.

The practical value of the booklet may be gauged from the fact
that it has now reached the 9th edition, this issue following closely
on the 8th edition which appeared only early this year.

Forthcoming Publications of the Council.

At the present time, the following future publications of the
Council are in the press:—

Bulletin No. 200.—" Preparation of Core Ingredients for Search-
light Carbons," by T. R. Scott, M.Sc., B.Ed.

Bulletin No. 201.—" Grazing Management: Continuous and
Rotational Grazing by Merino Sheep. 1. Study of the Production of
a Sown Pasture in the Australian Capital Territory under Three
Systems of Grazing Management," by R. M. Moore, B.Sc.Agr., Nancy
Barrie, B.Sc.Agr., and E. H. Kipps, B.Sc. *Appendix,* " The Measure-
ment of Pasture Yield under Grazing," by G. A. McIntyre, B.Sc.
" 2. The Effect of Continuous and Rotational Grazing on the
Infestation of Sheep with Internal Parasites," by H. McL. Gordon,
B.V.Sc., and Helen Newton Turner, B.Arch. " 3. Note on Pasture
Management," by J. Griffiths Davies, B.Sc., Ph.D.

Bulletin No. 202.—" The Strain Complex and Symptom Variability
of Tomato Spotted Wilt Virus," by D. O. Norris, M.Sc. (Agric.).

Bulletin No. 203.—" Agar in Australia," by E. J. Ferguson Wood,
B.A., M.Sc.

Bulletin No. 204.—" A Soil Survey of Part of Waterhouse Estate,
County of Dorset, North-East Coast, Tasmania," by G. D. Hubble,
B.Agr.Sc.

Bulletin No. 205.—" Studies on the Breeding Performance of
Ewes," by R. B. Kelley, D.V.Sc.

Bulletin No. 206.—" Pedogenesis Following the Dissection of
Lateritic Regions in Southern Australia," by C. G. Stephens, M.Sc.

Bulletin No. 207.—" The Fumigation of Wheat in Bag Stacks,"
by Frank Wilson and F. J. Gay, B.Sc., D.I.C.

Bulletin No. 208.—" Surface Fumigation of Insect Infestations in
Bulk Wheat depots," by Frank Wilson and A. T. Mills.

Bulletin No. 209.—" Interaction of Surface Infestation, Temperature, and Moisture Content in Bulk-Depot Wheat," by Frank Wilson.

Bulletin No. 210.—" Preliminary Survey of the Natural Pastures of the New England District of New South Wales, and a General Discussion of Their Problems," by R. Roe, B.Sc. (Agric.).

Bulletin No. 211.—" The Water Retting of Flax," by W. L. Greenhill, M.E., Dip.Sc., and Jean F. Couchman, B.Sc.

Bulletin No. 212.—" The Frictional Properties of Lead-Base and Tin-Base Bearing Alloys: The Role of the Matrix and the Hard Particles," by D. Tabor, Ph.D.

Bulletin No. 213.—" Laboratory and Field Tests of Mosquito Repellents," by R. N. McCulloch, B.Sc., B.Sc.Agr., and D. F. Waterhouse, M.Sc.

Bulletin No. 214.—" The Preparation and Properties of Synthetic Cryolite," by P. Dixon, M.Sc., and T. R. Scott, M.Sc.

Bulletin No. 215.—" Studies in the Biology of the Skin and Fleece of Sheep. 4. The Hair Follicle Group and its Topographical Variations in the Skin of the Merino Foetus," by H. B. Carter, B.V.Sc., and Margaret H. Hardy, M.Sc.

Bulletin No. 216.—" An Examination of the Peet-Grady Method for the Evaluation of Household Fly Sprays," by D. F. Waterhouse, M.Sc.

Bulletin No. 217.—" The Relative Importance of Live Sheep and of Carrion as Breeding Grounds for the Australian Sheep Blowfly *Lucilia cuprina,*" by D. F. Waterhouse, M.Sc.

Bulletin No. 218.—" Studies of the Physiology and Toxicology of Blowflies. 12. The Toxicity of DDT as a Contact and Stomach Poison for Larvae of *Lucilia cuprina:* 13. Insectary Tests of Repellents for the Australian Sheep Blowfly," by D. F. Waterhouse, M.Sc.

Bulletin No. 219.—" Spray Tests against Adult Mosquitoes. 1. Laboratory Spray Tests with Culicine (*Culex fatigans*) Adults," by D. F. Waterhouse, M.Sc. " 2. Spray Tests with Anopheline (*Anopheles punctulatus farauti*) Adults," by D. F. Waterhouse, M.Sc., and D. O. Atherton, M.Sc.Agr.

Bulletin No. 220.—" The Preparation and Use of Harvey's Reduced Strychnine Reagent in Oceanographical Chemistry," by D. Rochford, B.Sc.

Bulletin No. 221.—" Contributions to the Study of the Cell Wall. 4. The Nature of Inter-Cellular Adhesion in Delignified Tissue. 5. The Occurrence, Structure, and Properties of Certain Cell Wall Deformations," by A. B. Wardrop, M.Sc., and H. E. Dadswell, D.Sc.

Bulletin No. 222.—" The Chaetognatha of South-Eastern Australia," by J. M. Thomson, M.Sc.

Bulletin No. 223.—" Report of Marine Borer Survey in New Guinea Waters," by A. W. Shillinglaw, B.Sc., Dip.For., and D. D. Moore, B.Sc., A.S.T.C.

Bulletin No. 224.—" Mechanical Composition of Soil in Relation to Field Descriptions of Texture," by T. J. Marshall, M.Agr.Sc., Ph.D.

Bulletin No. 225.—" Studies on the Control of Wheat Insects by Dusts. 1. Field Tests of Various Mineral Dusts against Grain Weevils," by F. J. Gay, B.Sc., D.I.C., F. N. Ratcliffe, B.A., and R. N. McCulloch, B.Sc., B.Sc.Agr. " 2. Further Tests of Various Mineral Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C. " 3. The Use of Dust Barriers for the Control of Grain Insects," by F. J. Gay, B.Sc., D.I.C. " 4. The Use of DDT- and 666- impregnated Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C.

" The Commercial Timbers of Australia—Their Properties and Uses," by I. H. Boas, M.Sc.

" Handbook of Australian Pelagic Tunicates," by Harold Thompson, M.A., D.Sc.

A Note on Background Correction in Spectrochemical Analysis

By A. C. Oertel, M Sc *

Summary.

The density—log relative exposure curves, as obtained with a rotating stepped sector wedge and a Process plate, have the same general shape and the same gamma for monochromatic (line) radiation and continuous radiation (at the same wavelength) at least up to a density of 0.85 However, the curve for line plus background has a different shape and gamma at high densities from those of the curve for line alone. It is suggested that the differences are caused by a distortion of the relative exposure scale, the distortion being due to the fact that the background radiation is photographically effective only in the steps of large aperture. A method of approximate correction is given

1. Introduction

Agricultural samples are usually excited spectrochemically in the carbon or graphite arc, each of which emits a strong background of continuous radiation. In some parts of the spectrogram the lines of interest are so faint and the background is so intense that the lines can be detected visually only under favourable conditions of illumination of the spectrogram. For quantitive work it must be practicable to make a reliable measurement of the density of the faint line itself despite the much greater density of the background. The development of a spectrochemical technique for the accurate estimation of elements present at very low concentrations in agricultural samples involves the discovery of a simple and accurate method for correcting an apparent line density for the effect of the superposed background density.

A correction for background cannot generally be made by subtracting the density of the background from the density of line plus background, the two measurable quantities. Strock (3) found that the density of a line, as calculated by subtraction of the two measurable densities, increases more and more slowly and finally decreases as the exposure is increased (see curve D in Fig. 1). As Strock pointed out, the effect is due to the difference in the slopes of the density—log relative exposure curves for background alone and line plus background, although in the case illustrated by him the curve for the former has a smaller slope than the lower part, but a greater

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* An officer of the Division of Soils.

slope than the upper part, of the curve for the latter. Strock also found (4) that the slope of a density—log relative exposure curve derived from a narrow line was as much as 1·5 times greater than that of the curve derived from a continuous spectrum, and he showed that the difference could be explained as a consequence of the Eberhard effect. On the other hand Pierce and Nachtrieb (1), using Process plates, found that the curves for monochromatic and continuous radiation had the same gamma, and that there was no evidence of the Eberhard effect. They suggested that the results obtained by Strock may have been caused by the use of sensitized plates. Scott (2) apparently made no tests on the slopes of density-exposure curves for different radiations. His theoretical deduction of the basis for a method of background correction was based on the assumption that the slopes of curves for monochromatic and continuous radiations are the same, and an experimental test of the method showed that the assumption cannot be greatly in error.

2. Results and Discussion

The results obtained here show that (a) the density—log relative exposure curves are the same for monochromatic and continuous radiation at the same wavelength, at least up to density 0·85; (b) the curve for line plus background is different from that for line alone; and (c) the curve for line alone, obtained by subtracting background densities from line plus background densities, has a part in which increasing exposure apparently results in decreasing density. All spectra, line and continuous, were stepped by a rotating stepped sector wedge and photographed on Process plates. The curves plotted in Figs. 1 are not true H. & D. curves because intermittency effects and effects of failure of the reciprocity law are known to be present (the speed of rotation of the sector wedge was constant and about 750 r.p.m.). The curves are, however, those which are actually used in analytical work, and are accurately reproducible.

Spectrograms obtained included—

    (i) Line spectrograms, practically free from background, from a brass arc as recommended by Pierce and Nachtrieb (1).

    (ii) Continuous spectrograms from a graphite arc.

    (iii) Spectrograms of type (i) superposed on spectrograms of type (ii).

    (iv) Spectrograms of type (ii) superposed on spectrograms of type (i).

    (v) Line plus background spectrograms from an arc between graphite electrodes which had been thoroughly impregnated with a solution of brass in nitric acid and then dried at red-heat.

The density—log relative exposure curves for line plus background were obtained from spectrograms of types (iii), (iv), and (v), all of which consisted of a stepped line spectrogram plus a stepped background. All the curves were of the same general shape as curve B of Fig. 1. There was a small divergence at the upper ends of the curves caused by inaccuracy of measurement of high densities, by variations over the surface of a plate, and, it is suggested, by differences in density of backgrounds.

The curve for the line alone, obtained from spectrograms of type (i) is shown as curve A of Fig. 1. The original curve was moved bodily along the exposure axis until the point at density 0·5 coincided with the corresponding point of curve B. Curves A and B coincided over the whole of their lower parts and diverged at high densities, the curve for line plus background having the greater slope. It seemed possible that the density—log relative exposure relationship for line plus background differed from that for line alone.

All the background curves, from spectrograms of types (ii), (iii), (iv), and (v), were the same (curve C of Fig. 1), and agreed with the initial part of that for line alone. Therefore under the conditions obtaining, the density—log relative exposure relationship is the same for monochromatic radiation and for continuous radiation of the same wavelength, at least up to a density of 0·85, the highest background density obtained. That agrees with the result of Pierce and Nachtrieb. It also makes it improbable that the divergence of curves A and B is a real one.
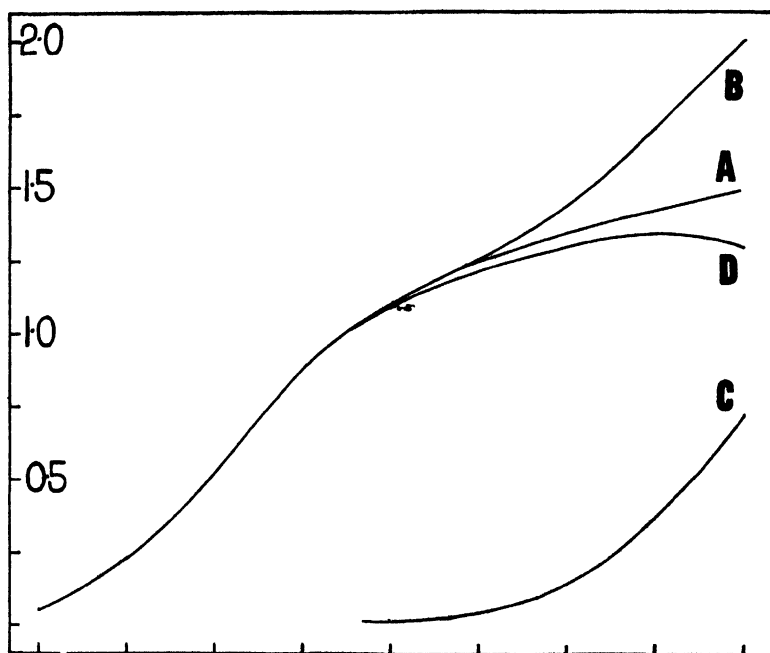

FIG. 1.—Typical density—log relative exposure curves.
    A. Monochromatic (line) radiation free from background.
    B. Line plus background radiation.
    C. Continuous (background) radiation at same wavelength.
    D. Corrected curve obtained by subtracting background from line plus background densities.

When the background densities were subtracted from the corresponding line plus background densities and the results plotted, density—log relative exposure curves were obtained, all of the same general shape as that of curve D. That curve agrees with the result

of Strock (3), and it is of interest to note that curve B has the same general shape as that of the corresponding curve obtained by Strock. The fact that curve D has a part in which increasing exposure apparently causes decreasing density makes it useless for analytical purposes. Nevertheless it agrees more closely with the curve for line alone than does the one for line plus background. A much closer agreement can be obtained by subtracting not the density of the background but a fraction of that density, the fraction being the ratio of the slopes of the upper straight part of curve B and the straight part of curve C. That fraction can often be obtained from the analytical spectrograms.

In the search for an explanation of the divergence of curves A and B, it is of interest that almost identical curves were obtained for line plus background from spectrograms of types (iii), (iv), and (v). It apparently does not matter whether background and line spectrograms are recorded simultaneously or consecutively, at least so far as practicable accuracy can distinguish. In the case of spectrograms of type (iii) the background radiation has been converted very inefficiently into density for the steps of small exposure (see curve C), while for the steps of greater exposure the conversion has been much more efficient and increasingly so. When the line spectrogram was superposed the steps of greater exposure had a start more than proportionately greater than that of the steps of small exposure. In other words the upper part of curve B was distorted with respect to the log relative exposure axis, the distortion increasing with increasing exposure. A similar explanation is satisfactory for curves derived from spectrograms of type (iv); but it is somewhat artificial for those derived from spectrograms of type (v) in which line and background are recorded simultaneously.

The density-intensity method of background correction (1), which has a sound theoretical basis, will not give curve A from curves B and C (that is, from the upper and lower parts of curve B). If it is accepted that curve B is not a true but a distorted density—log relative exposure curve, that result is to be expected. The test of the validity of the density-intensity method applied by Pierce and Nachtrieb was somewhat artificial in that a non-stepped background was superposed on a stepped line spectrogram. The uniform background produced very little distortion of the density—log relative exposure curve, the main effect being to shift the curve bodily parallel to the density axis. In this work stepped backgrounds were used because such backgrounds are necessarily obtained with stepped line spectrograms in analytical work.

The nearest approach to curve A that could be obtained by the application of the methods of background correction at present in use, theoretical or empirical (1, 3, 4), was curve D. A closer approach was usually given by the method suggested above, and if curve B is a distortion the method has a good theoretical basis. However, the method is not an entirely satisfactory one because the value of the fraction derived cannot be readily or accurately ascertained, the "straight" parts of the curves often having appreciable curvature.

The case of a very faint line on a dense background is more difficult of solution because the faint line itself records on the lower part of the density—log relative exposure curve where the increase in density with increased exposure is small, density measurements are difficult to make accurately, and the slope of the curve is changing rapidly. No simple method of correction has yet been found and further work is in progress.

3. Acknowledgment

Thanks are due to Miss Jean W. Burpee for technical assistance.

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The Influence of Cultivation on Soil Structure and Its Assessment in Soils of Variable Mechanical Composition

By G. B. Clarke, B.Sc.,* and T. J. Marshall, M.Agr.Sc., Ph.D *

Summary.

The change in water-stable aggregation, resulting from increasing periods of cultivation of grassland, was measured for two types of soil using a wet-sieving and a suspension procedure.

A significant decrease in aggregation was found in all cases. The major part of the decline which occurred over a period of up to twenty years took place in the first five years of cultivation. With respect to the initial levels of aggregation in the particular soils examined, the total decline in aggregation ranged from 42 to 69 per cent.

Regression equations relating aggregation and clay content were developed for sub-surface samples taken from the A-horizon below the depth of tillage. These equations were used as a basis for extracting the effect of mechanical composition from the aggregation data. The conditions are discussed under which this statistical device can be used for handling data from non-replicated plots where mechanical composition is variable. The use of multiple instead of linear regression equations, involving other fractions as well as clay, is also discussed.

The results obtained for water-stable aggregation by the wet-sieving and suspension procedures were based on limiting diameters of 0·2 mm. and 0·05 mm. respectively. The two methods gave parallel results with respect to the effects of clay content and cultivation upon aggregation.

Cultivation was found to have decreased the nitrogen content of the surface soil of both types significantly. There was not a highly significant effect below the depth of tillage. It is improbable that in these soils the decline in nitrogen content of the surface soil was the cause of the parallel decline in aggregation which resulted from cultivation.

1. Introduction

It is well established that cultivation reduces the water-stable aggregates originally present in uncultivated soil. Evidence for this is to be found in a number of comparisons made between cultivated soils with an extensive cropping history and soils under forest or grass. Usually a marked decline in water-stable aggregates has been demonstrated.

Much work has been done on prairie soils where reductions of 30, 27, 8 and 70 per cent. have been found by different workers (8, 4, 16, 3) in water-stable aggregates larger than 0·05 mm. The larger aggregates suffered even more severely from cultivation; aggregates larger than 0·5 mm. (4) were reduced by 87 per cent. and those larger than 0·1 mm. (3) by 75 per cent. in two different trials. A comparison (15) based on the surface area of aggregates has shown a similar decline of 85 per cent. In forest soils the magnitude of the decline is perhaps somewhat less than in prairie soils. Water-stable aggregates larger than 0·05 mm. declined by 40 and 20 per cent. in two trials (3, 16) and those larger than 0·02 mm. by less than 30 per cent. in most cases in a further investigation (1). As in

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. * An officer of the Division of Soils.

the prairie soils, a larger reduction was shown in the larger aggregates, a decline of 60 per cent. being noted (3) for aggregates larger than 0·1 mm. In a comparison based on surface area of the aggregates (15) a decline of 65 per cent. was noted. Investigations in Australia (7) and South Africa (11) on other types of soil have also shown marked reductions in water-stable aggregates following long periods of cultivation.

In all the cases discussed above, cultivation had been in progress for fifteen years or longer when the comparison with virgin (or approximately virgin) conditions was made. No indication of the rate of decline in water-stable aggregation is given by these results and not very much is known concerning this aspect. However, Civenko (6) has recently shown that, in successive years following the breaking up of " virgin turfed podzolic soil," the amount of stable structural aggregates larger than 0·25 mm. was 57, 50, 36, and 20 per cent. respectively.

In the present investigation, water-stable aggregates have been examined in soils which have been under a wheat-fallow rotation at the Waite Agricultural Research Institute for various periods of up to twenty years. It has been possible to draw conclusions concerning the progressive changes taking place under this cropping system in two types of soil.

Sufficient variability was found in the mechanical composition of the soils within each of the two groups of non-replicated plots to warrant attention being paid to the possible influence of this factor on the results. It is recognized that a greater development of water-stable aggregates is to be found in soils with a high than in those with a low content of clay. Baver (2) showed that the amount of primary particles smaller than 50μ present in water-stable aggregates larger than 50μ was related to the clay content of the soil. Rost and Rowles (16) have expressed the actual amount of aggregation found in this way as a percentage of the total amount of primary particles smaller than 50μ and have found that this relative expression of aggregation is also related to the clay content of the soil.

It follows that in making comparisons between the structural condition of soil samples from field plots, it will be desirable under some circumstances to apply a correction for the influence of mechanical composition on structure. A method was developed for applying such a correction using as a basis the relation between aggregation and clay content in samples taken from the A-horizon below the depth of tillage. In the present work it was found that clay accounted for a greater percentage of the variation in the actual than in the relative amount of aggregation. An expression for the actual amount of aggregation was used so that when effecting the correction for mechanical composition the residual variation would be at a minimum (see page 173).

The soils examined are within the red-brown earth zone in which the greater part of Australian wheat is produced. It is generally considered that cultivated soils in this zone are particularly susceptible to deterioration in structure (13).

2. Experimental

(1) *Description of Soils and Their Cultivation History.*

Two groups of four plots were available for examination at the Waite Institute. The plots had an average size of 0·2 acre. In one group, Type A, the A-horizon is a brown silty loam about 12 inches deep with some bleaching evident in the last 2 inches. The B-horizon is a light-brown clay loam to 24 inches and a red-brown clay below that depth with some lime occurring at about 30 inches. The C-horizon is colluvial material. In the second group, Type B, the A-horizon extends to an average depth of 10 inches and is a grey-brown clay loam or loam. The B-horizon is a red-brown clay with some lime at a depth of about 30 inches. This soil also has been formed on colluvial material. Soils of the red-brown earth zone to which these types belong have been described in detail by Piper (13).

Features of composite samples made up from the actual samples taken for structure measurements are listed in Table 1. The " surface " samples consist of soil down to a depth of 4 inches—the maximum depth of tillage; the " sub-surface " samples were taken below this and down to a depth of 8 inches unless any visible profile change occurred at a shallower depth.

TABLE 1.—MECHANICAL COMPOSITION OF SAMPLES FROM THE
A-HORIZON OF EACH TYPE.

Plot.	Treatment.	Number of Sample Sites	Depth (in.)	Mechanical Composition, Percentage Smaller Than—						Standard Deviation of Clay Content.
				5 mm.	2 mm	0 2 mm.	0 05 mm.	0 02 mm.	0 002 mm.	
Type A.										
A–0	Natural pasture	4	0–4	100·0	99·3	95·8	79·7	51·3	15·3	2·60
			4–8	96·8	95·1	92·9	77·9	52·9	19·3	3·12
A–1	Cultivated 1 year	8	0–4	99·1	98·3	94·0	78·5	52·9	19·0	3 17
			4–8	99·2	97·7	92·5	78·2	52·7	20·1	2·89
A–3	Cultivated 3 years	8	0–4	99·6	99·2	94·5	76·2	50·8	18·4	2·88
			4–8	99·5	98 6	93·4	76·8	51·8	19·9	3·83
A–20	Cultivated 20 years	4	0–4	99·7	98 9	95·5	77·4	50·7	18 2	0·75
			4–8	98·3	97·3	93·4	77·5	51·8	19·3	1·62
Type B.										
B–0	Natural pasture	10	0–4	99·4	97·9	93·7	81·3	57·1	22·1	6·97
			4–8	98·3	95·9	90·8	79·4	57·2	25·3	8·06
B–1	Cultivated 1 year	6	0–4	99·8	99·3	95·9	79·3	55·5	21·3	1·88
			4–8	99·9	99·6	96·1	80·8	58·6	27·1	4·39
B–11	Cultivated 11 years	4	0–4	99·7	98·8	94·4	80·5	56·5	22·1	3·31
			4–8	99·0	97·7	93·9	80·4	58·6	26·4	3·38
B–16	Cultivated 16 years	6	0–4	99·6	99·4	95·7	79·2	54·5	20·7	1·96
			4–8	99·1	98·9	95·7	81·6	57·7	26·0	7·04

Samples were collected from Type A just prior to a late fallowing at a time (October, 1944) when Plots A-1 and A-3 were in stubble following the crops of the previous year and Plot A-20 was in fallow. Samples from Type B were collected in August, 1944, when all the cultivated plots were under young wheat. An earlier preliminary sampling was also undertaken in January, 1944, on all the plots of

Type A. Surface and sub-surface samples were taken from each sample hole except in the case of the preliminary January sampling of Type A when only a limited number of sub-surface samples were taken for exploratory purposes. The number of samples taken from each plot varied with its size.

There was no record of any tillage on Plots A-0 and B-0 which were areas under natural pasture and trees adjacent to the cultivated plots. The remaining plots had been tilled annually under a rotation of fallow and a cereal crop in alternate years for the periods shown in Table 1. Wheat was the chief cereal grown but there were a few crops of oats. In one case only was any crop other than these grown. This was in the 10th year of Plot B-11 and the 15th year of plot B-16 when peas were grown in the fallow year and ploughed in. Natural pasture of long duration, with no known prior tillage, existed in all the cultivated plots up to the commencement of tillage, except for the 1-year and 3-years plots from Type A which had been under a sown pasture for ten years following an indefinitely long period of natural pasture.

(ii) *Laboratory Methods.*

The samples were air-dried, broken carefully by hand to pass a 1-inch sieve and sub-sampled by quartering. The mechanical composition was determined using a Bouyoucos hydrometer after mechanical stirring for 20 minutes using sodium oxalate as a dispersing agent.

Water-stable aggregates were determined in two ways—by suspension and wet-sieving procedures. In the first of these, the breakdown of aggregates by gentle dispersion is compared with the complete breakdown determined by mechanical analysis. Various devices for gentle dispersion are in use; that followed here involves mechanical shaking with water followed by the measurement of the amount of material in suspension with an effective diameter less than 50μ. Puri and Keen (14) have shown that there is an exponential relation between the amount smaller than about 2μ and the time of shaking. Such a relation was also found to represent the position for material smaller than 50μ.* After two or three minutes shaking the amount of dispersion still increased rapidly with increased shaking. It was only after about twenty minutes of shaking that reasonably steady values were obtained and the latter period was consequently chosen for subsequent work. A similar shaking period (fifteen minutes) was adopted independently by Downes and Leeper (7) because it was found to reveal differences better than other periods. The procedure used in this laboratory is similar in most other respects to that described by them.

A 50-gram sample was placed in a sedimentation cylinder after being wetted by capillarity overnight, and sufficient distilled water added to make the volume up to a litre. The cylinder, which was about 75 per cent. full, was shaken end-over-end for twenty minutes at the rate of eighteen revolutions per minute. An hydrometer reading was taken after a settling time of 40 seconds. This reading was compared with the 40 seconds reading of the mechanical analysis to obtain an expression for the water-stable aggregation. These readings are both taken to represent material with an effective

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* Unpublished data, 1937.

diameter smaller than 50μ. In the case of the mechanical analysis of these soils (which have a density of 2·65g./cc.) the representation is reasonably close. In the gentle dispersion procedure, however, some of the material will be in the form of small aggregates of density less than 2·65 g./cc. and these will be suspended with the primary particles in that portion of the suspension supporting the hydrometer. These aggregates will have a two-fold effect on the 40 seconds reading. In the first place, the presence of aggregates larger than 50μ (probably up to 70μ) having a settling velocity less than that of primary particles with the same effective diameter, will tend to increase the density of the suspension at the point of measurement. In the second place, the lower density of the aggregates will cause a lower suspension density than would be the case with the same mass of primary particles. To some extent these two effects compensate one another, but the latter source of error is by far the larger. Its magnitude will depend on the apparent density of the saturated aggregates and the ratio of aggregates to primary particles in suspension. In the comparative results reported here within individual soil types, this effect of aggregate density is not likely to be of much consequence.

A wet-sieving method developed by Meyer and Rennenkampff (12) with modifications introduced by Downes and Leeper (7) was the second method used in determining water-stable aggregates. In this method a sample is subjected to the disaggregating effect of water rising and falling through a fixed nest of sieves. The following procedure was adopted. A 25-gram sample was wetted overnight by capillarity and washed gently through a 5 mm. sieve on to a nest of sieves 3·5 inches in diameter with openings of 3, 2, 1, 0·5 and 0·2 mm. respectively. Water flow was commenced and was continued for twenty minutes with the filling and siphoning system controlled to give about 3·5 cycles per minute. The soil fractions remaining on the sieves were then dried in an oven at 105°C. and weighed.

(iii) *Expression of Results.*

The amount of particles smaller than 0·05 mm. present in water-stable aggregates larger than 0·05 mm. was determined by the gentle dispersion procedure. This will be referred to as " Aggregation (0·05 mm.) " and is given by the following expression:—

> Aggregation (0·05 mm.) = (Percentage particles smaller than 0·05 mm. by mechanical analysis) — (Percentage particles smaller than 0·05 mm. by gentle dispersion). (Percentages are calculated as percentage of total oven-dry soil.)

The amount of particles smaller than 0·2 mm. present in water-stable aggregates larger than 0·2 mm. was determined by the wet-sieving procedure and is referred to as " Aggregation (0·2 mm.)." This can be expressed as follows:—

> Aggregation (0·2 mm.) = (Percentage material larger than 0·2 mm. by wet sieving) — (Percentage particles smaller than 0·2 mm. by mechanical analysis). (Percentages are calculated as percentage of total oven-dry soil.)

Both these expressions give aggregation in terms of the amount of primary particles smaller than a given size which are present in aggregates larger than that size.

3. Water-Stable Aggregates

(1) *In Relation to Mechanical Composition.*

The sub-surface samples taken from below the depth of tillage provided data from which the following equations were drawn up relating aggregation and content of 2μ clay.

Type A—

Aggregation (0·05 mm.) $= -10·7 + 1·556$ clay, with $r = 0·845$ ($r = 0·517$ at $P = 0·01$) .... 1.

Aggregation (0·2 mm.) $= 23·7 + 1·611$ clay, with $r = 0·547$ ($r = 0·517$ at $P = 0·01$) .... 2.

Type B—

Aggregation (0·05 mm.) $= -8·5 + 1·216$ clay, with $r = 0·936$ ($r = 0·497$ at $P = 0·01$) .... 3.

Aggregation (0·2 mm.) $= 23·7 + 1·168$ clay, with $r = 0·740$ ($r = 0·492$ at $P = 0·01$) .... 4.

All four relations are significant at the 1 per cent. level. They hold within the range of clay content shown in Fig. 1. This diagram shows the relation between Aggregation (0·05 mm.) and clay content in sub-surface samples.
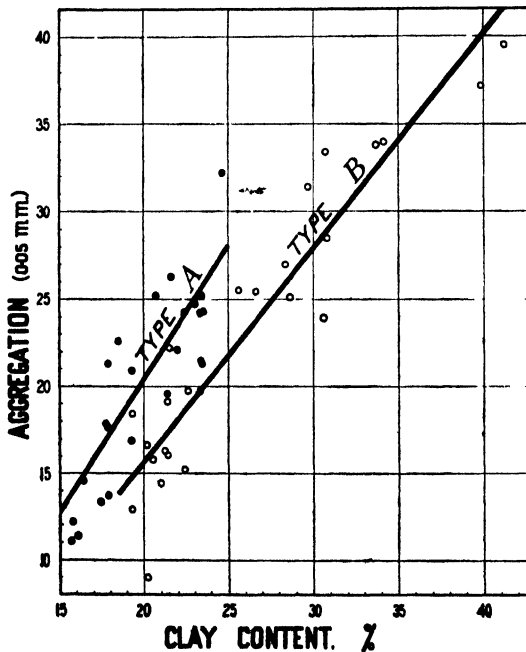

FIG. 1.—The relation between Aggregation (0·05 mm.) and clay content in sub-surface samples.

The effects of sand, silt, clay, and nitrogen content on aggregation of sub-surface samples were also studied by means of a number of multiple regression equations which will be discussed in a later paper.

These show that clay is predominantly the most active variable affecting aggregation in these soils and that although silt has a significant effect in some cases, the effect of mechanical composition on structure is reflected closely by the movement of clay content alone. No significant effect of nitrogen on aggregation was found in the sub-surface of either soil.

Regression equations could not be drawn up for the surface samples where the pronounced effect of cultivation masked the relation between aggregation and clay content.

(ii) *In Relation to Period of Cultivation.*

(a) *Aggregation data (uncorrected).*—Mean values for the aggregation of surface and sub-surface samples (with standard deviations) are given for each period of cultivation in Table 2.

TABLE 2.—WATER-STABLE AGGREGATION IN TWO SOILS AFTER VARIOUS PERIODS OF CULTIVATION.

Soil Type.	Period of Cultivation (Years).	Number of Samples	Surface.				Subsurface.			
			Aggregation (0·05 mm.).		Aggregation (0·2 mm.).		Aggregation (0·05 mm.).		Aggregation (0·2 mm.).	
			Mean.	S.D.	Mean.	S D.	Mean	S D	Mean.	S.D.
A	0	4	25·4	2·83	63·0	6·20	..	..	..	..
(Preliminary Samples.)	1	8	20·4	4·08	61·0	5·93	..	..	..	..
	3	8	15·7	4·00	52·7	5·88	..	..	..	..
	19	6	13·3	2·33	33·7	7·06	..	..	..	..
A	0	4	25·8	1·98	61·9	6·99	18·7	7·41	62·0	6·71
	1	8	19·6	4·75	52·2	6·51	20·5	2·77	56·2	7·94
	3	8	18·4	5·22	52·0	6·28	19·9	7·30	54·3	8·72
	20	4	12·5	0·55	40·6	2·25	20·9	5·29	51·9	11·31
B	0	10	22·8	5·34	54·2	7·74	21·0	9·81	53·8	10·28
	1	6	16·7	2.69	49·0	9·32	25·6	6·31	57·4	10·95
	11	4	14·7	2·54	41·7	8·14	24·8	3·67	52·1	8·99
	16	6	10·9	3·28	18·7	4·46	23·1	9·37	53·3	9·87

It appears from the uncorrected data in Table 2 that there is a well marked decline in aggregation of the surface soils with increasing period of cultivation. However, since differences between mechanical composition of the samples are considerable, it was considered undesirable to form conclusions without first extracting the effect of clay content. The sub-surface samples, relatively free from the influence of cultivation, do not reveal the trends shown by the surface samples. The question of the effect of cultivation on the sub-surface soil is discussed later.

(b) *Correction for the influence of mechanical composition in the surface samples.*—The influence of clay content on the aggregation of the surface samples was extracted from the data of Table 2 by means of the linear regression equations of aggregation on clay in the sub-surface samples (Equations 1 to 4). In order to apply this correction, it is necessary to assume that the coefficient of clay in the regression equations is the same for surface as it is for sub-surface

samples. It is also desirable that the sub-surface samples on which these equations are based should be relatively free from the effects of cultivation. These conditions are discussed further below.

The extraction is illustrated graphically in Fig. 2. Assuming the coefficient of clay to be the same in surface and sub-surface samples, it is possible to take the appropriate sub-surface regression line as a standard condition of aggregation in the surface samples to which trends in aggregation can be referred. For each sample there is a reference (or estimated) value of aggregation corresponding to its clay content. The difference ($\triangle$ aggregation) between the actual aggregation of a soil and the reference value can be measured along the ordinates on these diagrams. The departures from the reference basis are mainly positive in Fig. 2a but they become increasingly negative in Figs. 2b, 2c, and 2d respectively. This trend indicates a progressive decline in aggregation with increasing period of cultivation.
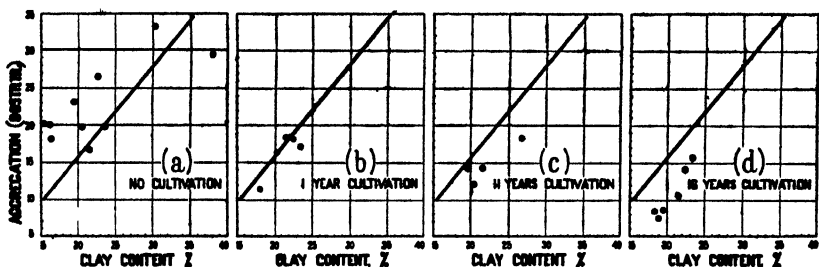

FIG. 2.—Relation of aggregation of surface samples of Type B to clay for four cultivation periods. The regression line (B of Fig. 1) is used as a reference basis.

The mean differences ($\triangle$ aggregation) between the actual and the reference values* of aggregation for Aggregation (0·05 mm.) and Aggregation (0·2 mm.) in Types A and B are given in Table 3 together with the results of anlysis of variance and t-test. In all four cases a significant decrease in aggregation due to the cultivation was found at the 0·1 per cent. level.

In general a decline in experimental error with cultivation was observed. This decline showed a positive linear relationship between the variance and the mean $\triangle$ aggregation per cultivation period and is probably due to the progressive effect of tillage machinery in smoothing out the variation in aggregation.

The mean deviations of the sub-surface data from the regression lines for each cultivation period are also of interest in connection with the influence of cultivation on aggregation in the sub-surface.

* Since no extensive sampling was done from the sub-surface of Type A at the time of the preliminary sampling, Equations 1 and 2 were used as the reference basis in that case. The regression equations might have been somewhat different for sub-surface samples taken at the time of the preliminary sampling because of the possibility of seasonal changes in aggregation (10). However, an error in the first constant of the equations would not affect the interpretation of cultivation effects, while an error in the second (the coefficient of clay) would do little other than increase the variance (see page 174).

In one case only, $\triangle$ Aggregation (0·2 mm.) in Type A, is a marked trend shown. An analysis of variance on this section of the data showed no significant effect over the four cultivation periods. A t-test on 0 and 1 year periods showed $t = 2\cdot03$ (at $P = 0\cdot05$, $t = 2\cdot25$). The influence of cultivation on this particular set of data is doubtless partly responsible for the low correlation, 0·547, in Equation 2. The fairly normal distribution of clay about plot means of similar magnitude, however, preclude a serious effect on the coefficient of clay in Equation 2.

TABLE 3.—RELATION BETWEEN AGGREGATION AND PERIOD OF CULTIVA-TION. ("$\triangle$ AGGREGATION" REPRESENTS THE DIFFERENCE BETWEEN THE ACTUAL AND THE REFERENCE VALUE OF AGGREGATION.)

Soil Type.	Period of Cultivaton (Years).	Surface.				Subsurface.			
		$\triangle$ Aggregation (0 05 mm).		$\triangle$ Aggregation (0 2 mm)		$\triangle$ Aggregation (0·05 mm.).		$\triangle$ Aggregation (0 2 mm.)	
		Mean.	S.D	Mean.	S.D.	Mean.	S D	Mean.	S D
A	0	12·1 ⎫	3·79	14·5 ⎫	7·27	..	..	..	..
	1	3·1 ⎬ a	3·75	8·3 ⎬ b	2·76	..	..	..	..
	3	−0·3 ⎪	4·17	1·4 ⎱ c	5·62	..	..	..	..
	19	−3·0 ⎭	2·18	−18·0 ⎰	7·66	..	..	.	..
A	0	12·7 ⎫	4·98	13·6 ⎫	9·33	−0·1	3·17	6·9	3·60
	1	0·7 ⎬ d	3·64	−2·3 ⎬ e	7·77	−0·3	3·37	0·0	5·81
	3	0·4 ⎪	2·93	−1·5 ⎪	4·91	1·5	2·25	−1·5	6·51
	20	−5·1 ⎭	0·67	−12·5 ⎭	1·39	−0·8	3·49	−3·3	11·38
B	0	4·1 ⎫ f	5·63	4·6 ⎫	9·77	−1·1	2·88	0·3	6·07
	1	−0·7 ⎬	1·43	0·3 ⎬ h	8·15	1·2	3·30	2·0	7·51
	11	−3·6 ⎬ g	2·06	−7·8 ⎪	5·28	1·3	1·40	−2·5	7·85
	16	−5·7 ⎭	1·16	−29·2 ⎭	2·86	0·1	2·32	−1·0	6·01

Summary of statistical analysis:—

> a. $P > \cdot001$ ($F = 13\cdot81$); b, $P > \cdot001$ ($F = 9\cdot03$); c, $P > \cdot001$ ($t = 4\cdot61$);
> d, $P > \cdot001$ ($F = 20\cdot71$)*; e, $P > \cdot001$ ($F = 13\cdot43$)*; f, $P < \cdot05$
> ($t = 1\cdot95$), variance ratio $= 15\cdot5$; g, $P > \cdot001$ ($F = 15\cdot32$); h, $P > \cdot001$
> ($F = 42\cdot63$)*.

By comparing the $\triangle$ aggregation of surface and sub-surface, it is seen that in all cases the surface of the virgin soils is better structured than the sub-surface. However, after the first year of cultivation this condition begins to be reversed.

The progressive deg[...] in water-stable aggregates in the surface soil is illustrated in F[...]3 where the data of Table 3 are plotted. In all but one case a[...] le[...] half of the total decline occurred in the first five years of cul[...]tion. The percentage of Aggregation (0·05 mm.) was reduced [...] 25·8 to 8·0 in Type A and from 22·8 to 13·0 in Type B. In [...] case of Aggregation (0·2 mm.), the corresponding decline was [...] 61·9 to 35·8 in Type A and from 54·2 to 20·4 in Type B. [...] losses in structure represent a total decline ranging

* Data transformed to square roots.

between 42 and 69 per cent. of the original virgin aggregation. The data from the preliminary sampling of Type A show declines which also fall within this range.
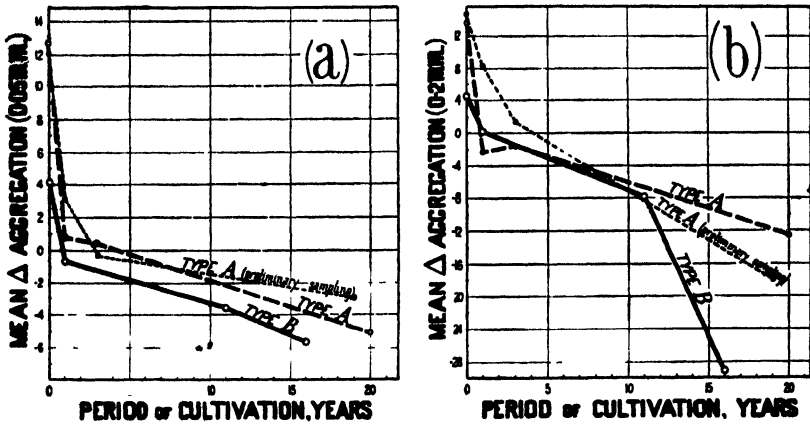

FIG. 3.—The effect of cultivation on water-stable aggregates in surface soil measured as *(a)* Aggregation (0·05 mm.) and *(b)* Aggregation (0·2 mm.).

4. Effect of Cultivation on Nitrogen Content

The effect of any progressive loss of organic matter with increasing period of cultivation is included with other factors (such as cumulative effect of rain action) that may cause the changes noted in aggregation. It is of interest to note the actual changes in nitrogen content which have occurred as a result of cultivation (Table 4 and Fig. 4). It can be seen from the diagram that, as is the case for aggregation, the decline in nitrogen content of the surface soil is greatest in the first few years of cultivation. Other work reviewed by Jenny (9) has shown similar trends. In these soils the ratio of carbon to nitrogen is about 12 (13), and nitrogen content can be taken as a reasonable guide to their organic matter status.
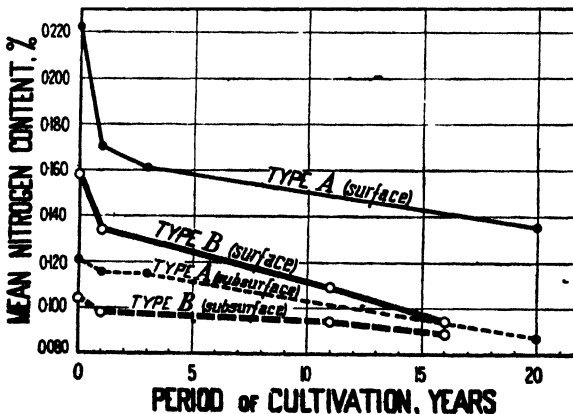

FIG. 4.—Relation between nitrogen content and period of cultivation for the surface and subsurface soils of Types A and B.

TABLE 4.—NITROGEN CONTENT* AFTER VARIOUS PERIODS OF CULTIVATION
(AS PERCENTAGE OF AIR-DRY SOIL PASSING A 2 MM. SIEVE).

Soil Type.	Period of Cultivation. (Years.)	Surface.		Subsurface.	
		Mean.	S.D.	Mean.	S.D.
A	0	0·222 }a	0·0345	0·121 ⎤	0·0266
	1	0·170 }b	0·0128	0·116 ⎬d	0·0120
	3	0·161	0·0073	0·115 ⎟	0·0120
	20	0·135 }c	0·0078	0·087 ⎦	0·0176
B	0	0·158 ⎤	0·0130	0·104 ⎤	0·0175
	1	0·134 ⎬e	0·0140	0·098 ⎬f	0·0108
	11	0·109 ⎟	0·0085	0·094 ⎟	0·0118
	16	0·094 ⎦	0·0105	0·089 ⎦	0·0152

Summary of statistical analysis:—

a, $P > ·01$ ($t = 3·93$); b, $P < ·05$ ($t = 1·73$); c, $P > ·001$ ($t = 5·67$); d, $P > ·05$ ($F = 3·93$); e, $P > ·001$ ($F = 39·38$); f, $P < < ·05$.

Results of statistical analysis of the data using analysis of variance and t-test are given in Table 4. They show a highly significant decline in the nitrogen content due to cultivation in the case of the surface soils but not in the sub-surface soils.

It will be noted that the level of aggregation in the surface and sub-surface of Type A is higher than the corresponding levels in Type B. A similar association was found in the nitrogen content levels of Types A and B. Furthermore the decline in aggregation due to cultivation is sympathetic with the decline in nitrogen content.

It was at first considered that nitrogen was responsible for the difference in the levels of aggregation and that the decline in aggregation was more immediately due to the decline in nitrogen content. However, multiple regression equations to be discussed in a subsequent paper show that there is no significant relation between aggregation and nitrogen content in sub-surface samples of either type.

In the surface of Type A, nitrogen content and Aggregation (0·05 mm.) are linearly related with a correlation of 0·863 and in Type B they are related with a correlation of 0·686. This correlation, however, has been induced by the influence of cultivation and therefore no conclusion can be drawn in regard to the influence of nitrogen on aggregation. Rost and Rowles (16) in discussing the difference between cultivated and uncultivated soil found a correlation between organic matter and aggregation; but observation of their data indicates that here also the correlation is a forced one.

It is probable then that, in these particular soils, both declines result independently from cultivation and that the decline in nitrogen content is not a cause of the parallel decline in aggregation.

* Analyses by Miss C. E. Moore, Soil Chemistry Section.

5. Discussion

The two methods used for assessing aggregation (wet-sieving and suspension procedures) gave much the same relation between aggregation and cultivation. Differences between means of aggregation for each period were of a larger order by the wet-sieving method, but on the other hand the variance was also greater. On the whole, the results (as shown in the diagrams) were more erratic by the wet-sieving method and it appears that a larger number of samples are required for use with this method than with the suspension method. The shaking period used here for the suspension method is longer than that usually adopted; and differences between treatment means and the magnitude of their variances would both have been greater had a shorter shaking period been used. Browning, Russell, and McHenry (5) have shown also that results by the two methods are closely related for samples from a given soil type.

The sympathetic relations of Aggregation (0·05 mm.) and Aggregation (0·2 mm.) to clay content within the one soil type are demonstrated by the regression coefficients of clay in Equations 1 to 4. The correlation coefficients indicate, however, that Aggregation (0·05 mm.) is more closely associated with the movement of clay content than Aggregation (0·2 mm.). An effect of this closer association lies in a greater percentage variation in aggregation being extracted from the surface data when Aggregation (0·05 mm.) is used to express structure. The use of Aggregation (0·2 mm.) leaves a larger variance error in the cultivation effects.

Cultivation has had much less effect on the sub-surface soil than on the surface as shown by trends in aggregation and nitrogen content. In the present data, where the clay content of the sub-surface soil is well distributed about the mean of each plot and where there are only small differences between plot means, cultivation effects would not in any case alter the coefficient of clay in the linear regression equations. They would however reduce the correlation coefficient and may alter the level of aggregation. These last two factors do not affect the application of the correction. With sharp differences between the mean clay content of different plots, these equations would lose their reliability only if there were also significant cultivation effects in the sub-surface. The sub-surface of the A-horizon should in many field experiments afford a useful basis for extracting the effect of mechanical composition from the aggregation of the surface soil.

The validity of the correction depends upon the assumption that the slope of the linear regression of aggregation on clay content is the same for the surface as it is for the sub-surface soils. For this to be the case the aggregating effect of primary particles of a soil must be the same in the surface and, sub-surface of the A-horizon. Any other variable affecting aggregation in the surface but not in the sub-surface is considered to be either a direct or indirect, effect of cultivation (or of virgin conditions).

Where another mechanical fraction, for example silt, has a significant effect on aggregation it will be shown in a later paper that aggregation can be. expressed as a linear function of clay and

silt. Where such a significant mechanical fraction exists the extraction of the effect of mechanical composition from the surface aggregation by the linear regression assumed for the surface may have two sources of error. In the first place a percentage of the variability in aggregation that could have been removed by a multiple regression would be left in the variance of the cultivation effects. In the second place a difference between surface and sub-surface relations of clay to the significant mechanical fraction may result in a difference in gradient between surface and sub-surface regressions of aggregation on clay. If the clay content in each surface plot is normally distributed about the same mean this error is one of variance only. On the other hand, if there are differences between each plot mean and the general mean, the mean differences in aggregation due to cultivation are not accurately determined by a linear regression. These sources of error are removed by using multiple regressions.

For these soils it has been emphasized that clay content is considered to reflect closely the effect of mechanical composition on aggregation. This is true even where other mechanical fractions exert a significant effect on aggregation because of the relations between clay and other mechanical fractions found in these soils. The use of the multiple regressions may be desirable when other mechanical fractions affect aggregation for the reasons discussed above but in these soils a correction by linear regression was found satisfactory.

One effect of extracting the influence of mechanical composition from aggregation by linear regression was to reduce the variance of the cultivation effects on aggregation. The second and most important effect was to reduce the error due to mechanical composition differences between plots. This correction should enable comparisons of structure to be made in soil types of local significance on which results from replicated plots cannot be obtained. In many cases there is sufficient variation in mechanical composition within one soil type to make such comparisons unreliable unless such a correction is applied.

6. Acknowledgments

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The Calcium/Manganese Ratio in Relation to the Growth of Lucerne at Canberra, A.C.T.

By E. H. Kipps, B.Sc.

Summary.

The unsatisfactory growth of lucerne on the savannah soil of Canberra, A.C.T., can be attributed to the high exchangeable manganese content of the soil in conjunction with an inadequate level of available calcium.

Raising the degree of base saturation of the soil and the pH to above 6·7, thereby reducing manganese availability to a minimum, permitted satisfactory growth. Additional increases in yield followed as the available calcium supply was augmented.

A correlation exists between the calcium/manganese ratio in the plant and the health and yield of the plants grown. Healthy plants had a Ca/Mn ratio greater than 66/1.

1. Introduction

Field observations on pastures of *Phalaris tuberosa,* lucerne, and subterranean clover indicated poor growth and vigour of lucerne (*M. sativa* L.) and of subterranean clover (*T. subterraneum* L.). The original observations were made in 1939 and the possibility of a trace element deficiency was recognized. In 1940 copper, zinc, manganese, and boron applied at appropriate rates gave no improvement in the growth of subterranean clover and/or lucerne in the pasture and no residual effects were present in 1941.

The symptoms are more marked in lucerne and have been described under both field and pot culture conditions by Shaw, Barrie, and Kipps (1944). In their pot culture study, lime at the equivalent of 1 ton per acre produced healthy and vigorous plants while plants receiving sodium molybdate at the equivalent of ½ and 2 lb. per acre made better growth than the unfertilized but were generally pale and yellow in colour.

It was apparent from these and other field and pot culture tests that the unsatisfactory growth of lucerne could not be attributed to a simple deficiency of the major nutrients—calcium, phosphorus, and potassium, or of the trace elements—copper, zinc, manganese, boron, and molybdenum. The possibility of sulphur deficiency had also been tested in the field, the addition of 56 lb. per acre of sulphur greatly reduced the yield of subterranean clover and *Phalaris tuberosa* in the pasture.

The unsatisfactory growth of lucerne was also found on the new Divisional Experiment Station at Dickson in 1941 on similar soils.

The Soils Division (Taylor, 1941) made a survey of the soils on the Dickson Experiment Station. The main body of the experiment station is covered by soils of common origin developed to their present form by differences of internal drainage and four stages in the catena can be defined. The well-drained member is the brown soil of the upper slopes, soil type 1, and the poorly-drained member is the grey depressional soil—type 4. Between the two extremes are a large

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* An officer of the Division of Plant Industry.

number of variants graded into one another and arbitrarily grouped into two intermediate types 2 and 3, varying in drainage capacity. The fifth type recognized is a typically alluvial fan. Some 70 per cent. of the area consists of soil types 1 and 2, and Taylor reports that no distinction need be drawn between them for field experiment purposes.

The soil used in the pot culture studies is the surface horizon, 0—5 in. from type 1, for which Taylor reports the following mechanical and chemical analysis:—

SOIL TYPE 1.

	Sample Number				
	6786.	6787.	6788.	6789.	6791.
Depth (in.)	0–5″	5–12″	12–18″	18–52″	52–72″
Gravel (per cent.) ..	Trace	5	8	9	Trace
Coarse sand	11·6	11·7	12·4	7·3	5·0
Fine sand	45·8	42·8	33·6	17·8	13·6
Silt	18·5	15·7	13·7	10·4	8·8
Clay	23·4	28·6	39·4	65·0	71·7
Loss on acid treatment ..	1·4	0·6	0·6	0·7	1·0
Nitrogen (N)	0·071	0·073	0·059	..	..
Phosphoric acid (P_2O_5) ..	0·058	0·053	0·065	.	..
Exchangeable base m.e. (per cent.)	5·0	..	8·3	11·0	..
Percentage composition of exchangeable bases—					
Ca	67	..	53	49	..
Mg	22	..	43	47	..
K	8	..	3	2	..
Na	3	..	2	2	..
pH	5·8	6·4	6·6	6·8	7·0

The total manganese content of the surface 0—5 in. layer of type 1 has been found by the author to be 0·126 per cent.; exchangeable manganese 0·019 per cent. The ratio of exchangeable Ca to Mn is 4·8 to 1. The relatively high content of both total and exchangeable manganese in the soil suggested that the calcium/manganese ratio played an important role in the poor growth of subterranean clover and lucerne on these soils.

2. Review of Literature

Several workers have reported high manganese content in soils. Robinson (1929) has quoted a range for U.S. soils from less than 0·001 to 1·27 per cent., and mentions that some tropical soils contain as much as 15 per cent. manganese.

Carr and Brewer (1923) reported little indication of manganese toxicity in Indiana soils when from 0·006—0·008 per cent. of soluble manganese was present, but 0·015—0·03 per cent. caused a very pronounced toxic effect.

Guthrie and Cohen (1910) reported unsatisfactory growth of *Cynodon dactylon* on small areas of a bowling green at Dubbo, New South Wales, where the affected soils contained 0·177 per cent. manganese. They also attributed poor growth of barley at Milton, New South Wales, and of wheat at Bathurst Experiment Farm, New South Wales, to excess total manganese. In all instances very patchy growth of the plants was noted.

McCool (1934) observed that the soluble manganese content of soils varied greatly with depth and with the time of the year, being lowest in autumn and spring.

The factors affecting manganese content of soils and vegetation have been reviewed by the Imperial Bureau of Soil Science (1940) and the results briefly summarized are as follows:—

(i) Cultivated soils usually contain less manganese than uncultivated soils.

(ii) The solubility of manganese in the soil depends largely on the soil reaction and the oxidation reduction equilibrium.

(iii) Manganese is one of the most easily exchangeable bases particularly under acid soil conditions.

(iv) Liming reduces the availability and solubility of manganese presumably as a result of oxidation.

(v) The manganese content of different plant species grown on the same soil varies considerably.

With regard to the uptake of manganese by plants, Svanberg (1938) reports a forage anaemia of horses, occurring in parts of Scandinavia, supposedly caused by Vitamin B_1 deficiency; Carlstrom and Hjarre (1938) noted the rapid disappearance of the vitamin from affected forage and suggested that this was probably due to its oxidation in the presence of large amounts of manganese. Svanberg states that the soil in affected areas is usually acid, with high sulphur and low calcium content, and suggests that the ratio of calcium to manganese in the soil may be important, as it ranges from 9·2/1 in unaffected areas to 1·8/1 in affected areas.

Wallace, Hewitt, and Nicholas (1945) studied soil acidity effects on runner beans and cauliflower and demonstrated that " the characteristic ' field acidity' symptoms of these crops, namely, intervenal chlorosis and necrotic spotting of runner bean, and incurled margins of cauliflower, are due to toxicity of manganese, though the toxic effect is considerably modified by calcium status, being particularly severe when calcium is at a low level, and substantially decreased by a high calcium status." By conducting suitable experiments these workers were able to establish the visual symptoms of calcium deficiency: " Runner bean—leaves slightly pale-green as for moderate nitrogen deficiency, with necrotic spots, especially near tips, around margin and progressing inwards inter-venally. Cauliflower—young leaves distorted, with tips brown and sharply hooked either backward or forward; older leaves marginal and intervenal areas becoming wilted and finally brown." The symptoms were stated to become more striking as the manganese level was raised.

Fried and Peech (1946) were concerned with gypsum and lime fertilization of lucerne and have stressed the inability of plants on acid soils to absorb calcium, even when supplied with an adequate quantity of soluble calcium salt. They state that there is good indication that manganese, which is readily absorbed by plants, may prevent the uptake of calcium and observed that the application of an additional 100 p.p.m. of manganese as carbonate to an acid soil virtually prevented the growth of lucerne. The percentage calcium in the plants was very considerably reduced by this treatment and the manganese content greatly increased, which suggests that antagonism occurs under conditions of extreme manganese availability.

Albrecht (1932) and co-workers have shown that the growth of legumes increases with the increasing degree of saturation of the clay with calcium, while Allaway (1945) has demonstrated that the availability of replaceable calcium increases with the increasing degree of calcium saturation of the soil.

3. Methods of Analysis

The oven-dried plant material (total above ground portion) was ground to pass a 40 mesh sieve and stored in air-tight containers. Nitrogen was estimated by the Kjeldahl method using $Se/FeSO_4$ catalyst. Calcium was determined on an aliquot of the HCl extract of the ash by the A.O.A.C. method.

Magnesium was determined on a further aliquot of the acid extract using 8-hydroxyquinoline after first removing the calcium as oxalate. Manganese was determined by the wet digestion method of Piper, and permanganate colour readings taken in a simple but sensitive single photocell colorimeter employing a d'Arsonval type galvanometer. Total and exchangeable manganese in the soil were also determined by the methods of Piper, using hydrochloric acid and ammonium acetate extracts respectively.

pH measurements were made on a $\frac{1}{2}$-in. core of soil removed from the pots, and suspended in water (1 to 5). Readings were made on a Cambridge glass electrode instrument.

4. Experimental

Experiment 1.

In the initial pot culture trial (Shaw *et al.*, 1944) it was found that healthy lucerne could be grown on Dickson soil by adding lime. Lime, however, can function in a dual capacity; firstly, by increasing the level of available calcium present in the soil, and secondly, by reducing soil acidity (pH effect). In order to determine, if possible, which of these effects was the more significant in this particular soil, a further pot experiment was carried out by Barrie and Kipps (1944, unpublished). The 0—5 in. horizon of soil type 1 (pH 5·8) was used with lucerne as the test plant. The plants were grown in 9-in. enamelled pots and the soil moisture readjusted to 22 per cent. on a dry soil basis at frequent intervals. The treatments applied were:—

Na_2HPO_4—an initial dressing of 1·018 g. per pot to all pots
(equivalent to 2 cwt. per acre).

Ca(OH)$_2$—0, 2·575, 5·15, 7·725, and 10·30 g. per pot (equivalent to 0, 5, 10, 15, and 20 cwt. per acre of Ca(OH)$_2$ on surface area).

MgO—0, 1·78, 3·56, 5·34, and 7·12 g. per pot (equivalent to 0, 5, 10, 15, and 20 cwt. per acre Mg(OH)$_2$).

CaSO$_4$—0, 5·99, 11·97, 17·96, and 23·94 g. per pot (equivalent in Ca to the Ca(OH)$_2$ treatments).

All levels of MgO and CaSO$_4$ were used in combination and four replicates of each treatment used.

The plants were harvested at the flowering stage and a total of six harvests made between January 20, 1944, and February 7, 1945. The yield of dry matter per pot, percentage nitrogen and calcium, and p.p.m. of manganese were recorded at each harvest.

Results.—It was found that magnesium hydroxide and lime produced similar increases in yield (Table 1). A further increase in yield was obtained with gypsum in combination with magnesium hydroxide, a maximum being reached in the vicinity of pH 6·7. The same pH was attained by lime or magnesium hydroxide alone and it would appear that the increase in the presence of gypsum can be attributed to the extra calcium, particularly as the soil is not especially low in sulphur (0·025 per cent. total, 0·012 per cent. sulphate sulphur).

Plants treated with gypsum alone gave the lowest yields and invariably appeared pale-green or yellowish in colour.

The manganese content was higher in all treatments than the normal (50-60 p.p.m.) quoted in the literature. Lime and magnesium hydroxide reduced the concentration in the plant, both alone and in combination with gypsum, the extent of the reduction depending upon the amount of base added to the soil. The highest manganese content was obtained in the final harvest of the nil treatment, this may be due to depletion of the available soil calcium. The addition of gypsum also caused a reduction in manganese at all harvests in spite of the lower pH (5·2) and consequently greater availability of that element in the soil.

TABLE 1 (see also Appendix A).—YIELD, PERCENTAGE CALCIUM, P.P.M. MANGANESE, AND SOIL pH.

—	Nil	Mg½	Mg1	L½	L1	G½ Mg½	G½ Mg1	G1 Mg½	G1 Mg1	G½	G1
Total Yield	203 7	263 1	286 2	255 1	274 5	348 4	328 8	361 4	354 4	173 2	167 1
pH	5 6	6 8	7 3	6 9	7 3	6 5	6 9	6 3	7 1	5 2	5·2
Mean Percentage Ca	1 19	1 08	1 00	1 44	1 50	1 37	1 09	1 39	1 23	1 42	1 44
Mean Mn p p m	323	167	103	166	130	179	146	181	124	191	277

G = gypsum, Mg = magnesium hydroxide, L = lime   ½ and 1 ton per acre

Experiment 2.

Unfortunately the highest level of acidity reached in the first experiment was no greater than that encountered in Duntroon soil in its natural state (i.e., pH 5·2). Although the usual mild symptoms were evident in unhealthy plants, there was a general lack of browning and withering of the leaflets. It was therefore decided to obtain further data on manganese distribution by extending the pH range on the acid side using the Dickson loam and pot technique adopted in the previous experiment.

Treatments.—All pots received an initial dressing of 5·25 g. KH_2PO_4 equivalent to 5 cwt. per acre 20 per cent. superphosphate and 2 cwt. per acre KCl.

$CaSO_4$. $2H_2O$ at 17·96 g. per lot (34·87 cwt. per acre $\equiv$ 8·11 cwt. Ca).

$Ca(OH)_2$ 7·725 g. per pot (15 cwt. per acre $\equiv$ 8·11 cwt. Ca).

Sulphur 3·34 g. per pot (6·49 cwt. per acre $\equiv$ S in $CaSO_4$).

NH_4NO_3 1·545 g. per pot (3 cwt. per acre).

The fertilizers were mixed throughout the soil in the pots about two months prior to planting. They were applied singly and in all combinations and three replications of each treatment were made.

Results.—The best yields on Dickson soil were obtained with the combinations lime plus gypsum, and lime plus gypsum plus nitrogen (Table 2). The plants were healthy throughout the duration of the experiment with the exception of a short period at the time of the fourth harvest when a slight paling of the leaves was observed.

Lime and nitrogen and lime alone gave similar results but in this case the period of paling was lessened.

All combinations in which sulphur was included produced unhealthy plants displaying a complete range of symptoms at one time or another and some mortality occurred in the absence of lime. The presence of lime enabled most plants to survive but growth and health were definitely poor.

The larger amount of gypsum applied in this experiment caused a correspondingly greater reduction in soil pH; the calcium content of the plants did not alter appreciably but the manganese uptake was much higher than that reported in the previous experiment. The plants were pale and yellow in appearance and some brown tips were observed on the leaflets, but all survived.

The addition of nitrogen caused an improvement in all cases, symptoms were less marked, and yields improved in its presence. Nitrogen exerted a marked effect in the presence of lime plus sulphur, and lime plus gypsum plus sulphur.

TABLE 2.—EXPERIMENT 2. DICKSON SOIL. (1945-6).

Treatment.

	No Calcium				Calcium (Gypsum)				Calcium (Lime)				Calcium (Lime and Gypsum)			
	Nil.	N.	S.	SN.	G.	GN.	GS.	GSN.	L.	LN.	LS.	LSN.	LG.	LGN.	LGS.	LGSN.
pH at— 16/11	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5	5·5
17/5	5·3	5·2	5·4	5·4	4·7	4·6	4·4	4·4	6·8	6·8	6·2	6·2	6·7	6·6	6·0	6·0
Total yield in grams	107·2	144·1	..	..	85·6	119·9	..	..	175·3	193·7	46·9	96·2	198·9	208·9	74·7	140·0
Per cent. Ca (Harvest Date)																
16/11	0·82	1·39	1·13	0·98	1·42	1·28	1·40	1·30	1·44	1·34	1·31	1·45	1·54	1·65	1·50	1·63
17/12	1·26	1·09	..	..	1·43	1·28	..	..	1·88	1·30	1·37	1·45	1·84	1·66	1·43	1·72
14/1	0·93	0·67	..	..	1·44	1·23	..	..	1·46	1·29	1·11	1·10	1·52	1·56	1·27	1·45
14/2	1·26	0·94	..	..	1·85	1·61	..	..	1·32	1·43	1·58	1·67	1·34	1·38	1·77	1·93
22/3	0·98	1·03	..	..	..	1·34	..	..	1·82	1·83	..	1·49	1·86	1·87	1·68	1·98
17/5	1·50	1·41	..	..	1·59	1·55	..	..	2·08	2·28	1·72	1·85	2·22	2·58	1·69	2·41
P.p.m. Mn																
16/11	190	450	..	2,600	300	430	2,500	2,700	180	190	2,400	1,000	190	210	1,400	500
17/12	225	320	..	..	240	360	..	..	160	190	1,850	1,120	205	200	1,060	440
14/1	260	280	..	..	480	490	..	..	140	180	1,600	1,130	250	150	1,280	460
14/2	520	790	..	..	1,160	580	..	..	275	270	4,600	1,670	310	200	..	190
23/3	230	400	..	..	..	400	..	..	100	150	..	550	170	130	3,000	130
17/5	230	320	..	..	240	360	..	..	170	200	700	450	160	190	490	210
Ca/Mn Ratio																
16/11	43	31	..	4	47	30	..	..	80	71	6	14	79	79	11	33
17/12	56	34	..	..	59	36	..	..	118	68	7	13	90	83	13	39
14/1	36	24	..	..	30	25	..	..	104	71	7	10	61	104	10	31
14/2	24	12	..	..	16	28	..	..	48	53	3	10	43	69	..	100
23/3	43	26	..	..	..	33	..	..	182	122	..	27	109	144	6	151
17/5	65	44	..	..	66	43	..	..	122	114	24	41	138	136	34	115

N = ammonium nitrate, S = sulphur, G = gypsum, L = lime,

Field Samples.—In December 1946, lucerne samples were collected in the field at Dickson and analysis of these plants indicated that 150-200 p.p.m. is the limit of manganese tolerance to be found in apparently healthy material in the field.

Description.	Mn. Content.
	p.p.m.
1 Normal plants (six year stand, S.W. corner) ..	81
2. Normal plants (six year stand, N.E. corner) .	137
3. Normal plants amongst chlorotic (second year)	98
4. Apparently normal (second year)	144
5. Apparently normal amongst chlorotic (first year) ..	196
6. Chlorotic plants (second year)	161
7 White necrotic lesions and brown tips (first year) ..	212
8. Prominent brown tips (first year)	225
9. Brown tips, older shoots only (six year stand) ..	296

Observations made on nil treatment pots in the preceding experiment prior to harvesting, confirm that plants appeared healthy below the 200 p.p.m. level, and thereafter as the manganese content increased showed chlorosis in varying degress of severity.

5. Discussion

Healthy plants and good yields of lucerne have been consistently produced on Dickson soil with a manganese content between 100 and 200 p.p.m. Both lime and magnesium hydroxide serve to reduce the manganese content of the plants to this level—the greater the addition of either base, the greater the corresponding reduction in manganese. However, as mixtures of lime and gypsum on the one hand and magnesium hydroxide and gypsum on the other produce higher yields than either of the bases alone, in spite of a higher resultant manganese content in the plant, it would appear that concentrations up to 300 p.p.m. are not necessarily detrimental to growth.

(i) *The Manganese/Iron Balance.*

The death of the plants growing under very acid conditions could be attributed to manganese toxicity or an upsetting of the manganese/iron ratio. Several rapid tests for iron were made on material from the first experiment and amounts of iron in excess of 1,000 p.p.m. were found to be present. It might be presumed therefore that sufficient iron was present to balance the range of manganese encountered in that experiment. In the second experiment in which manganese concentrations over 2,000 p.p.m. were observed it is improbable that the necessary iron was present to maintain the balance.

Steam sterilization of this soil produces extreme symptoms of manganese toxicity in beans, a four-fold increase in manganese taking place in the plant (from 500 to 2,000 p.p.m.) without any corresponding increase in total iron. In a sense iron might be considered to become " deficient " in respect to manganese under these conditions.

Twyman (1946) quoting the work of Somers and Shive (1942) states that " the theoretical explanation of the roles of manganese and iron to metabolic processes revolves round two facts; first, that the active functional iron is in the ferrous condition, and secondly, that the oxidation potential of manganese is higher than that of iron." Hopkins (1930) suggested that the presence of large amounts of manganese results in a high proportion of ferric ions or prevents the reduction of ferric iron.

In other words, the presence of manganese in more nearly normal quantities is a prerequisite to the formation of ferrous iron, and total iron determinations would then include a proportion of active ferrous iron; but in the presence of excess manganese, iron figures would mainly represent immobile ferric iron and become meaningless in so far as the manganese/iron ratio is concerned. The balance of active iron is apparently maintained within reasonably wide limits of manganese concentration, for in these experiments healthy plants have been grown containing many times the normal concentration of manganese when ample available calcium is present in the soil.

(ii) *Calcium Content.*

The results obtained show that the calcium content of the plant is dependent upon the calcium added to the soil. The mean percentage calcium rises with each increment of lime, and, to a lesser extent, with gypsum.

The application of magnesium hydroxide invariably reduces the percentage calcium content of the plant, but not necessarily the total amount removed from the soil. Above pH 6 the calcium uptake increases as additional gypsum is applied at any particular magnesium hydroxide level. The greatest uptake of calcium occurs at the $\frac{1}{2}$-ton magnesium hydroxide level around pH 6·4. Percentage magnesium in the plants on the other hand remains fairly constant in all treatments, and the beneficial effect of magnesium hydroxide apparently lies in increasing the degree of base saturation of the soil.

Comparing the yields obtained in the two experiments by taking an equivalent number of plants over a similar growth period, it is found that a mixture of lime ($\frac{3}{4}$ ton) plus gypsum ($1\frac{3}{4}$ tons) produces much the same increase in yield as a mixture containing magnesium hydroxide ($\frac{3}{4}$ ton) plus gypsum ($1\frac{3}{4}$ tons). The increase in yield may, therefore, be attributed to the greater availability of calcium resulting from an increase in the total base saturation of the soil.

(iii) *The Calcium/Manganese Ratio.*

Apart from a slight transitory paling in the fourth harvest, healthy plants were produced throughout the growing period in Experiment 2, when the following fertilizer treatments were employed: lime, and lime and gypsum, alone and in combination with nitrogen. The

remaining treatments were unhealthy and showed a range of symptoms including pale-green leaves, yellow leaves, brown tips, withering of the tips and edges of leaves, spindly and stunted growth of the entire plant.

The mild chlorosis of otherwise healthy plants at the fourth harvest is undoubtedly caused by the sudden rise in manganese content occurring at that period (Fig. 1). Fig. 1 also illustrates the modifying effect of lime and lime plus gypsum on the uptake of manganese by the plant.
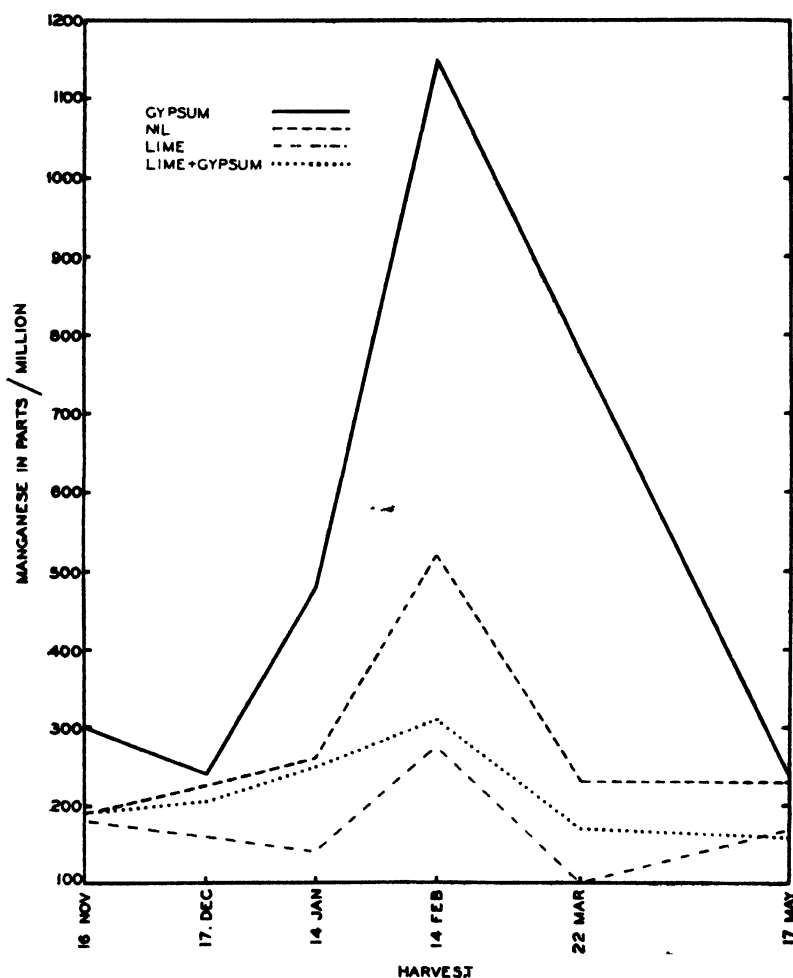

FIG. 1.—Variation in manganese content of lucerne grown in pots (Experiment 2).

The average calcium content of lucerne quoted in the literature is about 2 per cent. (Beeson, 1941), a figure which is higher than that obtained on most of the material grown in these experiments. It is generally recognized that the calcium requirement of lucerne is high,

and relative calcium deficiency in the soil may be the controlling factor in growth of this crop. The manganese content of the plants in this study is abnormally high and subject to wide fluctuations—increasing the calcium content of the soil by liming reduces the variability and amount of manganese in the plant; but the addition of gypsum to the soil increases the variability and quantity.

Leeper (1935) reports that manganese deficiency is confined to soils of pH 6·7 or higher, thereby inferring that manganese availability reaches a minimum at this stage. The results obtained in this study indicate that the highest calcium/manganese ratios are produced when the supply of available calcium is augmented in such a manner that the soil pH is of the order of 6·7. Fig. 2 shows that healthy plants contain at least 66 times as much calcium as manganese in their sub-aerial portions. As the ratio becomes less, pale-green and yellow symptoms appear at first, followed by the development of brown spots and the withering of the tips and margins of the leaves. Finally death occurs around the 5/1 level.
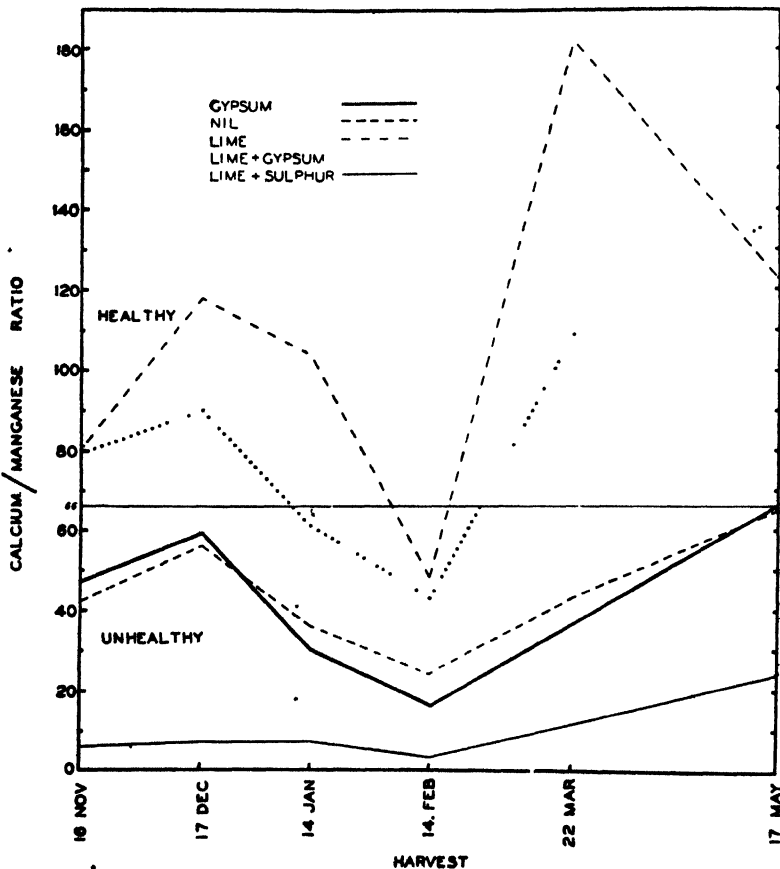
FIG. 2.—Variation in calcium/manganese ratio of lucerne grown in pots (Experiment 2). (Line is the top curve in the graph.)

6. Conclusions

The conclusions to be drawn from the two experiments are:—

1. The supply of available calcium in the soil is low for crops with a high calcium requirement such as lucerne. The "relative" deficiency of calcium probably accounts for the pale-green appearance of the crop.

2. The presence of large amounts of available manganese in the soil at certain periods of the year aggravates the already unhealthy condition of plants, resulting in browning and withering of the leaves.

3. Healthy and vigorous plants can be grown by adding lime or magnesium hydroxide to the soil at the rate of at least 15 cwt. per acre.

4. Maximum yields are obtained when the degree of base saturation is raised and an ample supply of available calcium is also present in the soil.

5. Maintenance of the above conditions over a prolonged period should lead to the establishment of a satisfactory stand of lucerne on this soil.

7. Acknowledgments

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Appendix A

EXPERIMENT 1.—THE EFFECT OF VARYING RATES OF LIME, MAGNESIUM HYDROXIDE, AND GYPSUM ON YIELD, pH OF SOIL, AND CHEMICAL COMPOSITION OF LUCERNE GROWN ON DICKSON LOAM.

(Yields in grams per four pots.)

	N11	CaSO₄ ¼	CaSO₄ ½	CaSO₄ ¾	CaSO₄ 1	CaSO₄ ⅛, Mg¼	CaSO₄ ⅛, Mg½	CaSO₄ ⅛, Mg¾	CaSO₄ ⅛, Mg1	CaSO₄ ¼, Mg¼	CaSO₄ ¼, Mg½	CaSO₄ ¼, Mg¾	CaSO₄ ¼, Mg1	CaSO₄ ½, Mg¼	CaSO₄ ½, Mg½	CaSO₄ ½, Mg¾	CaSO₄ ½, Mg1	CaSO₄ 1, Mg¼	CaSO₄ 1, Mg½	CaSO₄ 1, Mg¾	CaSO₄ 1, Mg1
Tons per acre																					
20/1/44 Yield	37.4	34.4	38.1	34.4	34.3	45.7	46.1	38.5	25.6	40.6	44.1	46.6	36.8	35.5	40.5	43.1	36.6	34.9	42.8	41.0	36.1
N	2.50	2.86	2.62	2.95	2.60	2.40	2.71	3.20	3.34	2.72	2.56	2.76	3.21	2.80	2.68	2.78	3.44	2.73	2.60	2.64	3.50
% Ca	1.28	1.41	1.34	1.50	1.06	1.12	1.43	1.07	1.27	1.12	1.13	1.00	1.05	1.10	1.47	1.12	1.20	1.29	1.17	1.13	1.06
ppm Mn	224	122	141	131	175	95	107	128	160	124		90	212	126	123		124	178	137	104	121
6/3/44 Yield	30.5	28.2	29.5	28.7	28.8	36.3	39.1	39.0	38.2	36.0	38.5	40.1	36.2	35.5	40.9	40.2	38.1	30.6	41.5	41.0	38.0
N	2.45	2.44	2.15	2.05	1.98	2.89	2.87	2.71	2.58	2.53	2.56	2.61	2.72	2.40	2.59	2.66	2.72	2.68	2.78	2.79	3.09
% Ca	1.24	1.43	1.38	1.51	1.30	1.24	1.16	1.14	1.07	1.29	1.44	1.14	1.07	1.37	1.25	1.18	1.18	1.40	1.24	1.33	1.26
ppm Mn	244		178	160	279	98		123	119	155	167	125	118	134	222	144	118	146	161	146	106
28/4/44 Yield	32.4	26.3	19.8	25.9	23.9	60.4	59.2	59.7	56.5	52.6	68.6	70.3	62.7	52.1	68.2	70.5	58.4	50.0	64.7	69.5	61.7
N	1.77	1.80	1.92	1.73	2.66	2.21	2.06	2.51	2.75	2.43	2.73	2.82	2.85	2.37	2.57	2.73	2.69	2.66	2.79	2.86	2.96
% Ca	1.28	1.51	1.42	1.54	1.26	1.20	1.10	1.15	1.15	1.54	1.42	1.36	1.13	1.57	1.70	1.32	1.17	1.53	1.38	1.37	1.29
ppm Mn	256	146	172	218	266	98	124	77	121	132	108	139	119	133		120	122	129	164	108	
8/44 pH	5.73	5.55	5.20	5.32	5.40	6.06	6.52	6.47	7.31	5.89	6.36	6.52	6.88	5.93	6.20	6.69	6.54	5.78	6.25	6.55	6.82
16/11/44 Yield	45.6	41.8	37.5	41.7	34.8	87.4	109.6	99.4	97.8	84.2	95.6	103.2	100.5	77.1	92.1	110.7	109.8	83.3	106.7	99.6	105.0
N	2.12	1.92	2.02	2.26	1.96	2.43	2.61	2.71	2.74	2.44	2.66	2.59	2.75	2.63	2.55	2.64	2.58	2.26	2.42	2.54	2.42
% Ca	1.21	1.77	1.66	1.80	1.71	1.36	1.36	1.38	1.27	1.63	1.74	1.52	1.34	1.86	1.73	1.46	1.50	1.58	1.75	1.65	1.54
ppm Mn	471	170	162	207	310	135	130	161	127	145	147	140	133	199	172	168	152	125	171	183	104
6/1/45 Yield	33.8	32.2	25.9	32.2	25.2	55.2	62.1	61.0	59.9	55.2	59.7	61.8	68.1	52.5	59.2	61.2	60.8	55.7	63.6	50.1	66.5
N	2.22	2.28	2.29	2.42	2.27	2.56	2.90	2.61	3.02	2.80	2.86	2.80	2.98	2.79	2.85	2.86	2.81	2.71	2.88	2.98	2.93
% Ca	0.99	1.31	1.34	1.40	1.53	1.14	1.01	0.96	0.93	1.18	1.21	1.15	1.03	1.23	1.34	1.14	1.17	1.26	1.28	1.40	1.14
ppm Mn	192	175	231	174	353	210	162	103	162	179	225	184	146	248	157	174	166	167	225	224	143
7/2/45 Yield	24.0	24.7	22.4	26.4	20.1	38.3	42.6	40.2	40.0	37.3	41.9	44.7	42.5	34.5	37.7	45.0	40.2	40.2	42.7	42.2	45.1
N	2.31	2.29	2.06	2.38	2.30	2.84	2.65	2.61	2.60	2.41	2.41	2.48	2.67	2.39	2.31	2.79	2.60	2.40	2.41	2.41	2.48
% Ca	1.18	1.50	1.36	1.54	1.80	1.17	1.13	1.10	0.99	1.33	1.29	1.20	0.91	1.29	1.31	1.06	1.19	1.17	1.28	1.21	1.08
ppm Mn	552	308	263	270	353	258	135	188	174	263	229	210	147	223	247	204	197	160	230	188	147
2/3/45 pH	5.54	5.57	5.15	5.22	5.04	6.28	6.64	7.04	7.35	6.40	6.57	6.85	6.91	6.13	6.70	6.76	7.04	5.99	6.43	6.94	7.45
Total Yield in grams	203.7	188.3	173.2	189.3	167.1	324.0	358.7	338.3	318.2	305.9	348.4	366.7	328.8	287.2	338.6	370.7	343.9	294.7	361.4	344.0	354.4
Mean % Ca	1.19	1.49	1.42	1.55	1.44	1.21	1.20	1.16	1.11	1.34	1.57	1.23	1.09	1.44	1.48	1.21	1.24	1.37	1.39	1.35	1.23
Mgm. Ca	2426	2770	2476	2983	2376	3963	4366	3950	3576	4245	4956	4611	3933	4277	5163	4666	4403	4138	5234	4827	4541
Mean % Mg	.58	.57	.52	.63	.60	.53	.61	.69	.71	.56	.61	.67	.75	.59	.63	.57	.73	.58	.99	.70	.51

Appendix A—*continued*

Tons per acre		Nil	Ca(OH)$_2$				Mg(OH)$_2$			
			¼	½	¾	1	¼	½	¾	1
20/1/44	Yield	37.4	35.6	42.1	43.4	43.0	41.2	43.0	28.9	12.1
	% N	2.50	2.46	2.60	2.63	2.73	2.55	2.76	2.50	3.77
	% Ca	1.28	1.63	1.40	1.12	1.34	1.17	1.03	1.25	1.27
	ppm Mn	224	136	134	148	108	150	121	162	
6/3/44	Yield	30.5	33.1	37.6	39.4	42.2	40.7	39.2	44.7	37.6
	% N	2.45	2.61	2.69	2.80	2.62	2.62	2.72	2.36	3.08
	% Ca	1.21	1.27	1.26	1.22	1.37	1.19	1.06	1.06	1.19
	ppm Mn	244	172		180	174	206	150	121	
28/1/44	Yield	32.4	45.1	54.6	52.9	55.2	48.7	53.6	45.9	45.5
	% N	1.77	1.97	2.23	2.20	2.42	1.84	2.20	2.07	2.02
	% Ca	1.28	1.51	1.37	1.34	1.43	1.60	1.02	1.09	1.10
	ppm Mn	256		130	135	114			130	105
8/44 pH		5.73	6.16	6.76	6.82	7.03	6.30	6.43	6.57	6.97
14/11/44	Yield	45.6	43.1	54.8	53.5	59.3	49.9	57.6	81.3	89.6
	% N	2.12	2.25	2.35	2.28	2.30	2.32	2.25	2.40	2.57
	% Ca	1.21	1.68	1.94	2.20	1.93	1.47	1.34	1.14	0.98
	ppm Mn	471	200	182	175	118	532	197	75	70
9/1/45	Yield	33.8	30.8	35.4	39.2	42.5	37.1	40.0	48.3	64.0
	% N	2.22	2.41	2.59	2.36	2.38	2.37	2.55	2.50	2.61
	% Ca	0.99	1.18	1.28	1.30	1.42	1.05	1.03	0.89	0.77
	ppm Mn	192	234	179	170	126	176	140	124	98
7/2/45	Yield	24.0	26.8	30.6	31.2	32.3	27.4	29.7	31.2	37.4
	% N	2.31	2.27	2.26	2.31	2.49	2.40	2.16	2.39	2.38
	% Ca	1.18	1.12	1.36	1.23	1.48	0.96	1.01	0.95	0.70
	ppm Mn	552	260	206	272	142	296	227	153	138
2/45 pH		5.54	6.68	7.05	7.52	7.58	6.76	7.01	6.98	7.61
Total Yield in grams		203.7	214.5	255.1	259.6	274.5	245.0	263.1	280.3	286.2
Mean % Ca		1.19	1.40	1.44	1.40	1.50	1.24	1.08	1.07	1.00
Mgm. Ca		2426	3070	3744	3748	4171	3089	2891	2949	2653
Mean % Mg		.58	.51	.54	.55	.53	.59	.68	.65	.68

The Treatment of Cut Potato Setts with Zinc Oxide

2. Infection of Stems and Tubers with *Rhizoctonia* and Scab

By J. G. Bald, M.Agr.Sc., Ph.D.*

Summary.

In this paper an analysis is made of the effects of treating cut potato setts with zinc oxide on stem lesions, the incidence of *Rhizoctonia* runner hyphae on the stems, and the incidence of scab and *Rhizoctonia* on the tubers.

Some treatments with zinc oxide and the organic mercury dip reduced the incidence of lesions and runner hyphae on the underground stems.

The zinc oxide treatments raised the yield of clean sound tubers.

Zinc oxide and mercury treatments gave partial or effective control of common scab.

The treatments that were effective against scab raised the incidence of *Rhizoctonia* sclerotia on the tubers.

Further investigation suggested an antagonism between *Actinomyces scabies* and *Rhizoctonia solani*.

It is suggested that the potato setts carried a surface microflora that grew with the shoots and on to the developing tubers, forming a sheath around the underground parts of the potato plant. The tubers after they were dug carried on their surfaces a microflora derived partly from the mother tuber and partly from the soil in which they were grown.

1. Introduction

The data of the trial described in this and the previous paper fall naturally into two sections, the first dealing with direct and indirect effects of treatment on the condition of the sett, growth, and yield; the second with infection of stems and tubers. The organisms causing infection were *Rhizoctonia solani* and *Actinomyces scabies* (and possibly others). These are ordinarily susceptible to control by treatment of the setts.

Two lots of infection data were taken, one at the same time as the setts were rated, the other after the yields were dug. The former consisted of records of lesions and macroscopically visible runner hyphae on the stems, the latter of scab lesions and *Rhizoctonia* sclerotia on the tubers.

2. Treatments

A list of the thirteen treatments in the trial is given below. Other details are presented in the previous paper (Bald, 1947). The treatments were:—

1. Clean tubers, cut October 22, setts dipped in water, put in small cloth bags, sufficient in each to plant one plot of the trial. The cloth bags were covered with damp sacks to maintain humidity until the setts were planted during the following two days, October 23 and 24.

2. Infected tubers, cut October 18, setts dipped in water, tipped into open trays and allowed to dry out. The setts lay two to three deep, the cut surfaces frequently in contact with the skin of other setts.

On October 22 they were put into small cloth bags, which were covered with damp sacks. A proportion of setts were definitely shrunken after the four days drying.

3. Infected tubers, dipped whole in water on October 18, left in trays until October 22, cut, bagged, protected by damp sacks.

4. Infected tubers, cut October 22, dipped in water, bagged, protected by damp sacks.

5. As for 2, but on October 18 dipped in zinc oxide suspension.

6. As for 3, but on October 18 dipped in zinc oxide suspension.

7. As for 4, but on October 22 dipped in zinc oxide suspension.

8, 9, 10. As for 2, 3, 4, but dipped in an organic mercury fungicide, improved Hortosan.

11, 12, 13. As for 2, 3, 4, but dusted with a zinc oxide-DDT dust.

The zinc oxide suspension consisted of 5 oz. zinc oxide in one gallon of water; the organic mercury dip was made up according to directions; and the dust contained 2 per cent. zinc oxide and 1 per cent. DDT in " pyrophyllite " filler. It was added to the setts in a barrel duster at the rate of 1 oz. to 8 lb. of setts, and the duster was turned until coverage of the setts was complete.

3. Estimation of Stem Infection

The sampling of plants and the examination of setts and stems were made on November 29, five weeks after planting, and twenty days after the first shoots were noticed above ground. The condition of the young plants at that time has been described (Bald, 1947). Lesions were clearly visible on the white and turgid stem tissues. They were of the type usually associated with *Rhizoctonia* injury—broad patches of slightly sunken necrotic tissue extending along the stem, sometimes girdling it. Some stems had been killed underground, forcing the development of shoots from axillary buds.

Where coarse runner hyphae were present, they could be seen quite clearly against the white stems. Generally, if they were present at all, they extended over the greater part of the underground stem. In many instances they had produced a few sclerotia; occasionally they formed the familiar weft of white mycelium above the ground level; sometimes they extended along the stolons and were associated with small lesions or killing of the stolon tips. On the harder tissues of the stems covered by runner hyphae there were often no lesions.

The impression given by the distribution of lesions and hyphae was that the faster-growing strains of *Rhizoctonia,* corresponding approximately to type C distinguished by Houston (1945), formed runner hyphae, sclerotia, and were barely pathogenic; others similar to Houston's type A, were slower-growing, much less likely to form runner hyphae or sclerotia, and were distinctly pathogenic.

4. Lesions and Hyphae on Underground Stems

(1) *Presentation of Results.*

On the plants dug for the examination of the underground parts were variable numbers of stems. This and small numbers of plants dug (two from each plot, 208 in all), probably increased the natural

variability of the plot samples examined for infection of the stems. A preliminary analysis of the numbers of stems per plant sampled revealed no significant differences, except between the two main blocks of the trial. The plants in the second block, situated in the more gravelly soil (Bald, 1947), had more stems than the plants in the block situated in the heavier soil. Treatment had no effect on the mean numbers of stems produced by a single sett. An analysis of covariance between numbers of stems carrying lesions and mean numbers of stems on the plants sampled revealed no consistent tendency for more infected stems to occur on plants with many stems than on plants with few.

As notes were taken of the extent and type of lesions on each stem, the results could have been presented in several ways. Finally, it was decided to present them as the mean numbers of stems with definite lesions (Table 1). These figures do not include a few stems that bore only occasional small lesions on the stolons, which were associated with the invasion of the underground parts by runner hyphae.

The numbers of stems coated with visible runner hyphae are similarly presented (Table 1). Treatment totals were modified according to the incomplete block design. This gave one negative value (treatment 5), but it was not significantly different from zero.

TABLE 1.—TOTAL NUMBER OF STEMS CARRYING (i) LESIONS AND (ii) VISIBLE RUNNER HYPHAE OF *Rhizoctonia*, ON 16 PLANTS SAMPLED FROM EACH TREATMENT. THE MEAN NUMBER OF STEMS PER PLANT WAS APPROXIMATELY THREE.

Description	Treatment No.	Numbers of Stems—			
		With Lesions.		With Runner Hyphae	
		Single Treatments	Means of Three Treatments.	Single Treatments	Means of Three Treatments
Clean setts, dipped in water	1	3	..	2·0	..
Infected setts, dipped in water	2	31	..	16·3	..
	3	20	..	9·3	..
	4	31	..	7·0	..
		..	27·3	..	10·9
Infected setts, zinc oxide dip	5	31	..	—2·1	..
	6	13	..	13·2	..
	7	15	..	4·6	..
		..	19·7	..	5·2
Infected setts, organic mercury dip (improved Hortosan)	8	23	..	7·1	..
	9	16	..	16·0	..
	10	5	..	0·2	..
		..	14·7	..	7·8
Infected setts, zinc oxide— DDT dust (pyrophyllite filler)	11	21	..	7·3	..
	12	14	..	20·8	..
	13	29	..	12·3	..
		..	21·3	..	13·5
Significant difference	..	14·3	8·3	10·6	6·1

(ii) *Controls.*

An obvious result, revealed in Table 1, was the relative absence of both lesions and runner hyphae on the stems from clean sound setts (treatment 1). By contrast, many stems from untreated infected setts (2, 3, 4) were damaged by lesions of the type usually attributed to *Rhizoctonia* solani; others carried runner hyphae and sclerotia of fast-growing strains of *Rhizoctonia.* The organisms responsible must have been mainly tuber-borne, not soil-borne.

There was in treatment 3 a lesser number of damaged stems, 20, than in treatment 2 or 4, 31. Although this difference was by itself not significant, there were similar differences in the other three treatment series. Altogether, there was a significant difference between the mean numbers of damaged stems from setts cut, then dipped or dusted, and allowed to dry out for four days (2, 5, 8, 11), and tubers treated whole at the same time, but cut four days later without wetting or treating the cut surface (3, 6, 9, 12). The crucial difference between these two series of operations was probably the treatment or lack of treatment of the cut surfaces, although they were not parallel in every detail from this one.

It was suggested in the section on the condition of the setts (Bald, 1947) that wetting (or dusting) the cut surfaces possibly encouraged their colonization by organisms carried on the skin of the tubers, even, under some circumstances, in the presence of protective substances. According to this interpretation, colonies of pathogenic organisms developing on the cut surfaces of the setts may later have invaded the young shoots, and increased the risk of damage to the underground stems.

(iii) *Zinc Oxide Dip.*

Two of the zinc oxide dip treatments, 6 and 7, reduced the production of lesions on the underground stems. Although in one of these treatments taken alone (6), the number of damaged stems was not significantly less than in the corresponding control (3), there was a strong suggestion of decreased infection, and 6 and 7 together were significantly less than 3 and 4. Treatment 5, in which the cut surfaces of the setts were allowed to dry out after dipping, caused no reduction in the number of damaged stems.

The effect of the three zinc oxide dip treatments on the subsequent invasion of the underground stems by runner hyphae was different from their effects on the organisms causing lesions. The fast-growing strains of *Rhizoctonia* that produced runner hyphae and sclerotia seemed to have readily invaded the unprotected cut surfaces of the setts (6) without the encouragement of extra moisture, and later to have spread up the young shoots. On the other hand, the zinc oxide dip gave protection against these strains of *Rhizoctonia* when it covered the cut surfaces (5, 7), even when the surfaces were allowed to dry out after dipping (5).

This differential effect on the two types of organism may have been due to differences in their powers of invading tuber tissue. The fast-growing surface-inhabiting forms of *Rhizoctonia* could not readily escape contact with the zinc oxide when it covered the whole surface of the setts, and their growth must have been checked continually

by its action. The slower growing pathogenic strains, given a sufficient duration of favourable conditions, such as those produced by wetting the cut surfaces of the setts, might penetrate the surface cells and remain active, even though suberization may have cut them off from the underlying tissues of the setts.

(iv) *Organic Mercury Dip.*

There was very little evidence either of lesions or runner hyphae on the stems from setts treated with the mercury dip on both skin and cut surfaces, the day before planting began (treatment 10). This treatment was useless as a practical measure of control, because of its serious effects on growth (Bald, 1947), but it demonstrated that a complete cover of an efficient fungicide might almost eliminate the production of stem lesions due to infection from the sett. If tubers had been treated and planted whole, efficient control of stem lesions would probably have been attained.

Neither of the other two mercury treatments produced a significant reduction in the number of damaged stems, presumably because the cut surfaces were not in a condition to resist invasion by fungi capable of causing lesions on the stems.

The different effects of the three mercury treatments on the subsequent development of runner hyphae were similar to those of the zinc oxide dip treatments.

(v) *Zinc Oxide-DDT Dust.*

The amount of damage to stems arising from setts treated with the zinc oxide-DDT dust was, in one important point, similar to the effects of those treatments on the setts themselves. There was evidence that the zinc oxide-DDT dust as a whole might have an action contrary to that expected of the zinc oxide component. This is evident from the results of treatment 13, in which the dust was applied to the cut surfaces the day before planting began, and the setts were kept in a humid atmosphere until planting. A thick coating of dust stuck to the surfaces and became damp by the withdrawal of water from the exposed and ruptured cells. In spite of the zinc oxide in the dust, a favourable culture medium for organisms that later produced lesions on the stems was apparently established and maintained on the tuber tissues beneath the dust.

Whereas in treatment 13 the number of stems with lesions was greater than in the parallel zinc oxide dip treatment (7), treatment 12, in which the dust was not put directly on the cut surfaces, kept the value for damaged stems at the same level as the corresponding zinc oxide dip treatment (6). The value for treatment 11 instead of being greater was less, though taken alone not significantly less than that for the corresponding zinc oxide dip treatment, 5, and control treatment 2. It was however, low enough to demonstrate an opposite tendency in the action of the dip and the dust. Treatment 11 and the corresponding treatments 2 and 5, may be compared from this point of view with treatment 13 and the corresponding treatments 4 and 7. It was noticed that the dust on the cut surfaces of the setts in treatment 11 encouraged rapid dehydration of the surface tissues. Thereby it discouraged suberization, but it may

also have discouraged the establishment of organisms, as much by its drying action as by the presence of zinc oxide. Fewer organisms may later have resulted in fewer lesions on the stems of plants in treatment 11.

Compared with the zinc oxide dip, dusting favoured the strains of *Rhizoctonia* that produced runner hyphae. The total of stems with runner hyphae was, as a whole, significantly higher in the dusting treatments, 11, 12, 13, than in the zinc oxide dip treatments, 5, 6, 7. The tendency already observed for a higher value, following the mode of application in which the cut surfaces were not treated (12), was again apparent. Probably the zinc oxide in the dust had some repressive action on the development of the faster-growing surface-inhabiting forms of *Rhizoctonia,* which in part counteracted the effects of the filler-DDT components of the dust.

5. Yield of Clean Sound Tubers

To obtain an unbiased estimate of the effects of treatment on the incidence of scab and other defects of the tubers, sorting and weighing was done by farm hands without supervision. The grade standards were set beforehand. They were:—

1. Over 3 ounces, clean and sound, reasonably good shape, no more than one or two small superficial scab lesions on any tuber, but a higher tolerance for *Rhizoctonia* sclerotia than is allowable in the best commercial grades.

2. Similar to 1, but poor shape.

3. Over 3 ounces, infected with scab or severe *Rhizoctonia,* cracked, distorted by spotted wilt infection, injured mechanically, or damaged by larvae of the potato moth.

4. All tubers under 3 ounces.

In the final analysis of yields, grades 1 and 2 were bulked, that is, the effects of treatment on the yields of clean sound tubers over 3 ounces were examined. The results are in Table 2. Because of the

TABLE 2.—YIELD OF CLEAN SOUND TUBERS, GRADES 1 AND 2 COMBINED.

Treat-ment	Yield	Treat-ment	Yield	Treat-ment	Yield	Treat-ment	Yield	Treat-ment	Yield.	Mean Yield.
		2	23·6	5	29·3	8	22·3	11	24·0	24·8
		3	18·1	6	25·2	9	26·5	12	29·3	24·8
1	22·4	4	23·9	7	28·9	10	23·4	13	29·0	26·3
Mean yield			21·9		27·8		24·1		27·4	
Significant difference between two treatment totals			..		..		..			5·56

different origin of the setts in treatment 1, the comparison of the yield with the yields for other treatments is not valid. Treatment with zinc oxide dip raised the yield of clean sound tubers on the

average by 27 per cent. One of the zinc oxide-DDT dust treatments (11) failed to give an increase over the corresponding control (2), because the total yield was lower. As a whole, the dust treatments also gave a significant increase in yield of clean sound tubers. Of the mercury dip treatments, 8 and 10 equalled the corresponding control treatments, 2 and 4, in spite of much lower total yields. The other mercury treatment gave a significant increase.

Of the nine treatments, six gave increases that were clearly or probably significant, and the other three were on the same level as the corresponding controls. All nine treatments gave a higher percentage of clean sound tubers over 3 ounces than the corresponding controls.

6. Scab Lesions on the Tubers

A method of estimating the extent of scab infection on the tubers was evolved for this trial. It was a variation of the method of Clark *et. al.* (1938) for the estimation of the percentage of tuber surface covered with lesions. Several lots of tubers, lightly infected, moderately and severely infected, were rated tuber by tuber in comparison with the illustrations of percentage tuber surface covered with lesions, given in the paper quoted above. Also, two dimensions of each tuber, length and breadth, were measured. The product of these two dimensions was used to weight the rating for each tuber, and to give an approximate correction for the size of the tuber. The sum of the products for individual tubers, percentage surface covered with scab lesions $\times$ length of the tuber $\times$ breadth, was divided by the sum of the products, length $\times$ breadth. This gave an estimate of the percentage of the whole tuber surface of the sample covered by scab lesions.

Standards were thus established, using samples such as are obtained from infected plants, each consisting of a number of tubers with various amounts of scab mixed with scab-free tubers. This provided a visual scale for comparison with plot yields. The plot yields were spread out on a floor after they had been graded, each plot thus being represented by four grade lots. The percentage of tuber surface covered was estimated, by comparison with the standards, for each of the four grades of each plot yield. As there were 104 plots in the trial, the amounts of scab injury to 416 lots were estimated. A single value for each of the 104 plots was obtained by multiplying each grade rating by the weight of tubers in that grade, adding the four products, and dividing by the total plot weight. The 104 values so obtained were submitted to a full analysis. The analysis itself and previous trials showed the method to be consistent and reliable.

The treatment means and the minimum significant difference between two treatment means are in Table 3. The percentages of tuber surface covered in the three infected controls were between 4 and 5, and all other treatments were significantly lower. Some treatments gave partial control, others effective control of scab. Of the 9 dipping and dusting treatments, 5 were not significantly different from the controls planted with clean seed (treatment 1); these all gave partial control. They included two zinc oxide dip

TABLE 3.—PERCENTAGE OF TOTAL TUBER SURFACE COVERED BY SCAB LESIONS.

Treat-ment.	Per-cent.	Treat-ment.	Per-cent.	Treat-ment.	Per-cent.	Treat-ment.	Per-cent.	Treat-ment.	Per-cent.	Mean Percent.
		2	4·24	5	1·61	8	·40	11	1·04	1·82
		3	4·99	6	2·35	9	1·12	12	1·46	2·48
1	2·12	4	4·50	7	·78	10	·52	13	1·89	1·92
Mean percentage		4·58		1·58		·68		1·46		

Significant difference between two treatment means .. .. .. | 1·04

treatments, 5 and 6, one mercury dip treatment, 9, and two zinc oxide-DDT treatments, 12 and 13. In treatment 1 itself, there was a significant amount of scab, suggesting either soil-borne infection, or infection carried without visible lesions on the surface of the setts. The latter appears the more likely explanation, as two mercury treatments, 8 and 10, gave effective control of scab (0·40 and 0·52 per cent. of surface covered). It appears unlikely that the treatment of the setts would give any protection to the tubers of the plants growing from the setts.

The toxic effects of the mercury dip on the cut setts, and the resulting reduction of the leaf area and loss of yield, in practice, would make such treatments as 8 and 10 useless. The zinc oxide-DDT dust treatment, 11, also gave excellent control of scab, but caused a reduction in growth and total yield. The amount of scab in treatment 7 (0·78 per cent.) was not significantly greater than in the most effective mercury treatments, and emergence, growth, and total yield were not adversely affected.

Comparing the percentage cover by scab lesions in the thirteen treatments with the percentage yield of clean sound tubers, an association between treatment means was clearly evident (Fig. 1). There were, however, irregularities in the association, which arose from the fact that scab was not the only reason for rejecting tubers from grades 1 and 2. Also, there was a general tendency for the higher yielding treatments to produce lower grade tubers, which further reduced the association.

Because of the many variables in this trial, which might not be present or effective under other conditions, the most reliable estimate of the value of any treatment against scab is probably given by the total yield and the percentage cover of the tuber surface with scab lesions. By these criteria, the most effective control of scab was obtained from dipping the cut setts in zinc oxide suspension the day before planting began, and keeping them in a humid atmosphere until planting (7). The next most effective was treating whole tubers in the organic mercury dip, and cutting them the day before planting (9). The next was treating whole tubers with zinc oxide-DDT-pyrophyllite dust, and cutting before planting (12).

A difference of theoretical interest, evident in the treatment means, is between the methods of applying treatments to the setts. On the whole, the treatments in which the cutt surfaces were treated (2, 5, 8, 11 and 4, 7, 10, 13) gave better control of scab than those in which the whole tubers were treated before cutting and the exposed surfaces were left unprotected until planting (3, 6, 9, 12). The results are in the last column of Table 3.
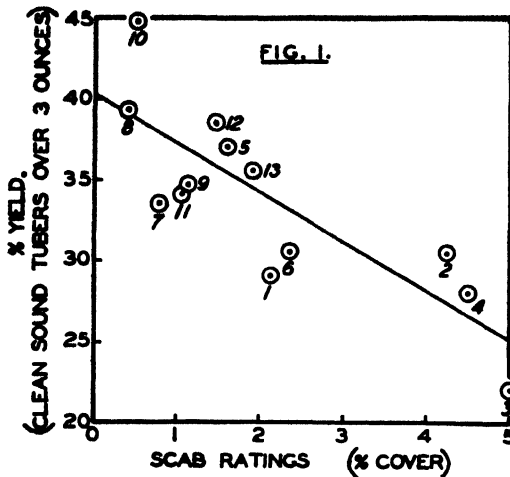
FIG. 1.—Percentages of total yields consisting of clean sound tubers over 3 ounces in each of 13 treatments, plotted against percentages of tuber surface covered by scab lesions.

There is an interesting comparison, also, between these values and the numbers of stems on which *Rhizoctonia* runner hyphae had developed 5 weeks after planting. The figures corresponding to those in the last column of Table 3 are 0·89, 1·85, 0·75. Where the cut surfaces were exposed after treatment, receiving no direct application of water, dip, or dust (3, 6, 9, 12), infection was higher than when the cut surfaces were also treated. Presumably, both the strains of Rhizoctonia that produced visible runner hyphae and sclerotia, and the *Actinomyces* causing scab grew rapidly over the surface of the developing plant, and found the unprotected cut surfaces of the setts excellent sites for the establishment of colonies. Both extended from the setts, presumably up the stems, and then along the stolons to the developing tubers. It will be shown later that this type of *Rhizoctonia* probably also invaded the young tubers from the soil, and produced sclerotia on them when they were mature, completely swamping the effects of invasion from the setts. This was not true of the *Actinomyces* causing scab lesions, which, in this trial, must have arisen predominantly from the setts.

7. Rhizoctonia Sclerotia on the Tubers

The estimation of *Rhizoctonia* on the tubers was made on an arbitrary scale. A rating of 10 was given to a sample of tubers 100 per cent. infected with a moderate coating of sclerotia. If about 20 per cent. were so infected, the rating was 2. The rating was

increased or decreased according to the number and size of sclerotia on individual tubers. Although the method was arbitrary, it was found, after standards were set, that two people rating the same lots independently recorded similar values. The method was reliable enough for the estimation of the incidence of *Rhizoctonia* in this trial.

The mean ratings for *Rhizoctonia* sclerotia on the tubers are in Table 4. A value for each plot was obtained by rating the four grades independently, and combining the four grade ratings as for scab. The 104 values so obtained were analysed in full.

TABLE 4.—MEAN RATINGS FOR INFECTION OF TUBERS WITH *Rhizoctonia sclerotia.*

Treat-ment.	Rating.	Treat-ment.	Rating	Treat-ment.	Rating.	Treat-ment	Rating	Treat-ment	Rating	Mean Rating.
		2	2·21	5	2·78	8	4·70	11	2·74	3·11
		3	2·13	6	2·85	9	3·98	12	3·56	3·13
1	2·16	4	2·05	7	3·52	10	3·76	13	2·91	3·06
Mean Rating			2·13		3·05		4·15		3·07	

Significant difference between two treatment means   0·72

Treating the setts, instead of reducing the incidence of *Rhizoctonia* sclerotia, increased it. Most of the increases from treatment were unmistakably significant. The greatest infection was in the mercury treatments, where the least might have been expected. Only one zinc oxide dip treatment, 5, and one zinc oxide-DDT dust treatment, 11, failed significantly to increase the incidence of *Rhizoctonia* sclerotia on the tubers.

The *Rhizoctonia* sclerotia and associated runner hyphae on the tubers were similar in type to those previously found on the stems of the young plants sampled 5 weeks after planting, and it was natural to look for some association between the numbers of infected stems in each treatment and the *Rhizoctonia* ratings of the tubers dug from the plants in those treatments. No association was found between treatment means.

This type of *Rhizoctonia* appears to have been a normal inhabitant of the soil, and to have invaded the developing tubers from the soil. As the tubers expanded, the mycelia extended over them, and ultimately produced sclerotia, thus masking the effect of the initial infection. There were indications in the treatment means that, if the sclerotia due to infection could have been estimated and eliminated from the total ratings, an association would appear between the numbers of the stems with runner hyphae sampled after 5 weeks, and the residues of tuber infection with *Rhizoctonia* sclerotia.

An underlying association may be illustrated by crude graphical methods, based on a negative association between scab and *Rhizoctonia* which will be illustrated in the next section of this paper. The latter is assumed to give a measure of the sclerotia arising from soil-borne *Rhizoctonia*. By estimating the soil-borne infection for each treatment and subtracting it from the treatment total, an estimate of the sclerotial infection arising from the setts may be obtained. These estimates could have been made by the objective use of partial regressions, but uncertainties that might still exist in the interpretation of the results hardly seemed to justify the labour of calculation.

A freehand curve was drawn as accurately as possible through the graph of *Rhizoctonia* ratings plotted against percentages of tuber surface covered by scab lesions (Fig. 2). The curve was not linear.
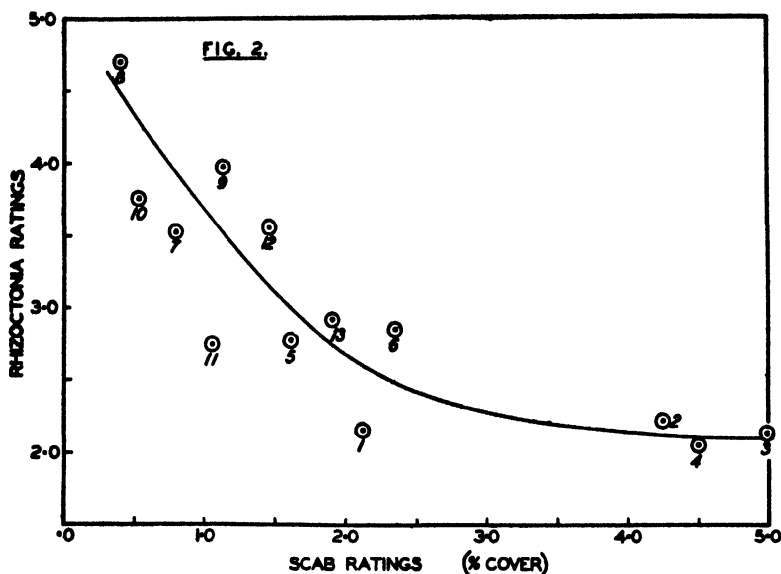
FIG. 2.—Relation between *Rhizoctonia* ratings of tubers from 13 treatments, and percentages of tuber surface covered by scab lesions. A freehand curve is drawn as accurately as possible through the points

Instead of attempting to obtain directly the residual *Rhizoctonia* ratings, representing sett-borne infection, the residuals were taken in the form of positive or negative deviations above or below the curve in Fig. 2. These residuals (Table 5) were plotted (Fig. 3) against the corresponding treatment means for numbers of stems, examined on November 29, carrying visible runner hyphae (Table 1).

Although these methods are not fully objective and the interpretation of the results is open to question, there is no doubt about the association between the quantities plotted in Fig 3. It suggests that runner hyphae of fast-growing strains of *Rhizoctonia* may in many instances have grown from the setts up the stems of young potato plants, along the stolons and on to the tubers. There they produced characteristic sclerotia as the tubers ripened.

TABLE 5.—RESIDUAL *Rhizoctonia* RATINGS REPRESENTING SETT-BORNE INFECTION. DEPARTURES ABOVE OR BELOW THE CURVE DRAWN THROUGH THE GRAPH OF *Rhizoctonia* RATINGS ON SCAB RATINGS FOR THIRTEEN TREATMENTS.

Treat-ment.	Rating.	Treat-ment.	Rating.	Treat-ment.	Rating.	Treat-ment.	Rating.	Treat-ment.	Rating.	Mean Rating.
		2	·09	5	− ·27	8	·23	11	· ŗ2	·04
		3	·03	6	·36	9	·46	12	·42	·32
1	− ·45	4	− ·06	7	− ·43	10	− ·56	13	·14	− ·23
Mean Rating			·02		− ·11		·04		·23	
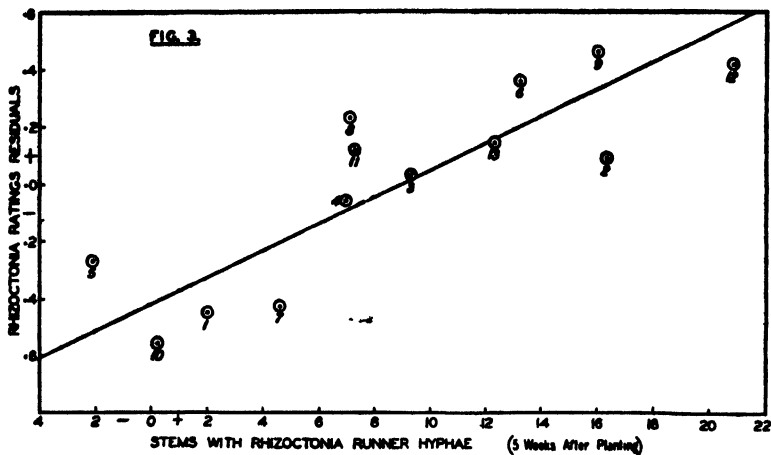
FIG. 3.—Residues of ratings for infection of tubers with *Rhizoctonia* sclerotia, after elimination of components presumably due to soil-borne infection, plotted against numbers of stems coated with *Rhizoctonia* runner hyphae amongst those examined 20 days after emergence began.

8. Negative Association between *Actinomyces* and *Rhizoctonia*

(i) *Between Treatments*.

It was obvious when the plot yields were laid out for rating that where the incidence of *Rhizoctonia* was high that of scab was generally low, and vice-versa. This negative association is illustrated in the treatment means (Tables 3, 4). Using the 26 treatment means within blocks, a correlation coefficient was calculated for the correlation between scab percentages ánd *Rhizoctonia* ratings. The value obtained was − 0·6374, which was significant. This value is an underestimate of the association, as, in calculating the correlation coefficient, linearity was assumed for the relation between scab percentages and *Rhizoctonia* ratings, whereas the true relation appears to have been curvilinear (Fig. 2.).

There was a possibility that this association was due to environmental conditions favouring one or other disease in restricted areas of soil, but a more likely explanation was an antagonism between *Actinomyces* (or some associated organisms) and *Rhizoctonia*. It has been suggested that the growth habits of the two were similar, both extending rapidly over the surface of the growing plant, and that the majority of the *Rhizoctonia* sclerotia were derived from mycelium invading the young tubers from the soil. Presumably, where the *Actinomyces* invaded the tubers first, it suppressed the development of invading *Rhizoctonia*.

This implies that the *Actinomyces* causing scab, or some organisms closely associated with it, extended over much greater areas of tuber surface than were actually covered by scab lesions. On the average, less than 5 per cent. of the tuber surface was covered with lesions, even in the most severely affected treatments. If the *Actinomyces* were the only organism antagonistic to *Rhizoctonia,* and were present only in the lesioned areas, more than 95 per cent. of the surface would have been subject to invasion by *Rhizoctonia,* and little evidence of antagonism would have been obtained.

(ii) *Between Ratings of Single Tubers.*

Further evidence of antagonism between *Actinomyces scabies* and the strains of *Rhizoctonia* producing sclerotia was sought from the incidence of the two diseases on single tubers. It seemed unlikely that two sets of contrasting environmental conditions would exist around individual tubers. The persistence of a negative association at this level would be evidence of antagonism.

There was already some evidence from the yields of single plants, that the negative association held for units of that size, but that it might be complicated by the effects of treatment. To prevent any serious complicating effect of treatment, the sample of single tubers examined was taken from the bulked grade 3 yields of treatments 2 to 4. Each tuber was rated for both scab and *Rhizoctonia*. For scab, the illustrations of percentage tuber surface covered by lesions (Clark *et al.,* 1938) were used as the standards of comparison. For *Rhizoctonia* an arbitrary scale, 0 to 6, was used, 0 representing freedom from sclerotia, and 6, numerous relatively large sclerotia over the whole surface of the tuber. The intermediate ratings, 1 to 5, represented increasing sizes and concentrations of sclerotia. Where a relatively large proportion of the surface was covered with pitted scab lesions, only that part of it free from scab lesions was considered in estimating the concentration of the sclerotia.

A random sample of 175 tubers was examined and rated individually. The results are in Table 6, which is in the form of a contingency table. The distribution of frequencies suggested a negative association between the incidence of the two diseases. Where there were numerous scab lesions, the sclerotia as a rule were relatively few and small, and vice versa. The significance of the negative association was tested by calculating chi-squared; the value obtained was 58·83, which, for 12 degrees of freedom, was highly significant. There was in, this good evidence for the hypothesis of antagonism between *Actinomyces* (or closely associated organisms) and the strains of *Rhizoctonia* that produced runner hyphae and sclerotia.

TABLE 6.—CONTINGENCY TABLE, TWO WAY CLASSIFICATION OF SINGLE
TUBERS ACCORDING TO RATINGS FOR SCAB AND *Rhizoctonia*.

Rhizoctonia Rating.	Frequencies.	Scab—Percentage Surface Covered·					
		0.	1-3.	4-9.	10-27	28-100	Total.
0	Actual	4	10	11	15	22	62
	Expected	16·30	12·40	9·92	12·40	10·98	
	Difference	−12·30	− 2·40	1·08	2·60	11·02	
1	Actual	5	12	8	13	5	43
	Expected	11·30	8·60	6·88	8·60	7·62	
	Difference	− 6·30	3·40	1·12	4·40	− 2·62	
2	Actual	18	7	5	3	3	36
	Expected	9·46	7·20	5·76	7·20	6·38	
	Difference	8·54	− 0·20	− 0·76	- 4·20	− 3·38	
3, 4, 5	Actual	19	6	4	4	1	34
	Expected	8·94	6·80	5·44	6·80	6·02	
	Difference	10·06	− 0·80	− 1·44	− 2·80	− 5·02	
Total		46	35	28	35	31	175

Chi-squared = 58·83, P < 0·001.

(iii) *Examination of Scab Lesions and* Rhizoctonia *Sclerotia on the Tubers.*

A number of tubers was examined under a hand lens with a magnification of 20. The tubers were wetted and held in the sun; under the lens the runner hyphae of *Rhizoctonia* were clearly visible. These hyphae formed a weft of mycelium between the sclerotia, and were widespread over the surface of infected tubers. Very often the hyphae ran along cracks in the skin of the tubers, and possibly deepened them by killing adjacent superficial cells. When the hyphae were very numerous, and their action relatively severe, sufficient cells were killed to produce a silvery appearance. On such areas there might be many minute sclerotia, each formed from a few hyphae. Zones of this kind were generally found at the stem end of the tubers, suggesting that, in these cases at least, invasion of the young tubers had occurred by way of the stolons.

The weft of *Rhizoctonia* mycelium was often thickest and most vigorous in the eyes of the tubers and covering the dormant buds. When the growing tips emerged, they often lifted the web of hyphae with them. Examination with binoculars at a higher magnification showed continued growth of the mycelium. Sometimes the presence of active hyphae was associated with small lesions on the leaf primordia folded over the growing point; until the point itself was attacked, growth continued. The hyphae associated with these lesions on the growing tip were not as coarse or dark as the typical runner hyphae.

In spite of such growth habits, when *Rhizoctonia* runner hyphae reached the edge of a scab lesion, which was nearly always of the pitted type, they seldom descended into it; they turned aside and skirted it. Often the hyphae did not even penetrate between adjacent scab lesions, but skirted the intervening tissue as well as the lesions

themselves. Sometimes sinuous runner hyphae were seen near and in scab lesions, superficially they had the same appearance as the exhausted hyphae sometimes found in culture. No sclerotia were seen in fully developed pitted lesions, even on tubers where there was a zonal distribution of scab and *Rhizoctonia*.

Occasionally on the surface of a tuber were lesions which appeared to result from the interaction of the two organisms. They had the appearance of superficial or slightly raised scab lesions, consisting of a circle of more or less rectangular spots or freckles arranged like the numbers on a clock face. Along the radiating lighter-coloured areas were often *Rhizoctonia* runner hyphae. The impression gained from examining a number of them was that they were initially scab lesions, checked at an early stage of their development by the invasion and antagonistic action of *Rhizoctonia* hyphae. They sometimes took on a concentric or irregular form, or merged into small zones of freckling that had lost the appearance of a distinct lesion. They were similar to the type 4 scab lesion illustrated by Clark *et al.* (1938).

9. Discussion

The example described in this paper, of antagonism between two components of the microflora of potato tubers, was probably not the only one. The microflora on the underground portions of the potato plant seemed to be made up of components from both the sett and the surrounding soil. Throughout the data, gathered in this trial, on sett rot, stem lesions, runner hyphae on the stems, scab lesions, and *Rhizoctonia* sclerotia on the tubers, there were indications of positive and negative associations between the various microfloral components. These associations were relatively weak, and correlation coefficiencies might reach the 1 in 20 level of significance only when values for one or more of treatments 1, 8, 10, or 11 were eliminated from the calculation. However, in the mass, they created an impression of interrelated and antagonistic development that was suggestive of a definite microfloral population on the surface of the underground parts of the potato plant.

The more effective treatments were shown to reduce the *Actinomyces* that were carried on the sett and that finally extended to the developing tubers and produced pitted scab lesions on them. Probably also it affected the strains of *Rhizoctonia* carried on the setts that were capable of forming sclerotia on the tubers. If there were saprophytic elements of the microflora, not macroscopically visible, that normally grew along the stems and stolons on to the developing tubers, their growth also would be more or less suppressed. The result of treating the setts would be equivalent to a partial sterilization of the surface of the young plant, and the consequences might be a radical change in the ultimate constitution of the surface microflora of the plants because of invasion of organisms from the soil. If the soil contained organisms pathogenic to the potato plant, treatment of the setts could lead to increased infection by a reduction of the natural defences of the plant, the saprophytes capable of antagonistic action against soil-borne pathogens.

A logical conception of plant protection arising from this picture of a microflora would be a method of destroying pathogens on the potato sett selectively, leaving saprophytes capable of an-

tagonistic action unharmed. If that were impracticable, an alternative would be to destroy or suppress the whole microflora of the setts, and substitute one saprophyte or more capable of antagonistic action against soil-borne pathogens.

Attempts to use antagonistic organisms for the suppression of plant pathogens have been made, e.g., Millard and Taylor (1927) added cultures of *Actinomyces praecox* to sterilized soil inoculated with *A. scabies,* and thereby reduced the *A. scabies* population to negligible proportions. When *A. praecox* was added to unsterilized soil infected with *A. scabies,* it failed to exert this protective action (Goss, 1937). Of such attempts Garrett (1944) writes:—

" The special conditions favouring the development of antagonism, or parasitism of one microorganism on another, on the agar plate or in sterilized soil are therefore absent in unsterilized or natural soil. This may be concisely expressed by saying that *natural soil is biologically buffered.* For this reason " biological control " has often failed to fulfil in field trials its extravagant promise on the agar plate or in pots of sterilized soil."

Probably the potato setts and shoots are also " biologically buffered." Suppression of the population of the microorganisms on the setts that are responsible for the buffering action, reduces or eliminates it (see above); but this also might be used as the first step in introducing another population of non-pathogenic microorganisms on to the setts, particularly on to the cut surfaces. It might be possible to select organisms from the normal surface microflora of the potato plant, or produce them by cultural methods, that are resistant to zinc oxide, or to some other efficient fungicidal or fungistatic agent. If such organisms were found, potato setts might be treated with a mixture of the chemical agent and cultures of the resistant and antagonistic saprophytes. The organisms might not need to be antagonistic in the sense that they caused actual damage to the pathogenic organism; they might need only to be strongly competitive. These organisms, freed of serious competition from the original microflora of the sett, would then have to be capable of rapid growth over the surface of the emerging shoot, and of forming a protective coating on the underground stems, stolons, and tubers. They would be present on the plant to buffer it against invasion by soil-borne pathogens. They would need to have a wide range of adaptability if they were to be efficient in a wide range of soils.

In this trial, infection of the tubers with *Rhizoctonia* sclerotia arising from the soil was of interest, because, as far as is known, the patch of soil in which the trial was planted had never grown potatoes before. Probably the strains of *Rhizoctonia* producing sclerotia were present as normal constituents of the soil microflora, and were capable of surviving in a clean, well-cultivated fallow.

Finally, in a summary of the efficiency of the treatments in controlling the diseases of the stem and tuber, number 7 again appeared somewhat better than the others (Bald, 1947). It reduced the numbers of lesions on the stems to the same level as did the mercury treatment, 9, which was the closest to standard practices for the control of such diseases. Under the conditions of the trial, the reduction

of lesions on the stem gave no improvement in growth or condition of the plants; but field experience is that under other conditions, such lesions might spread and cause damage to the growing haulms. It seems likely, although it has yet to be proved, that treatment with zinc oxide might be a useful method of reducing *Rhizoctonia* injury to the growing plant.

Treatment 7 reduced the invasion of young stems by fast-growing runner hyphae of the less pathogenic strains of *Rhizoctonia*. In a soil not carrying a high population of such strains, this might result in some reduction of sclerotia on the tubers. It gave excellent control of scab when scab was causing a reduction in yield of the better-grade tubers. The anomalous action of nearly all treatments on infection of the tubers with *Rhizoctonia* sclerotia was shared by treatment 7 in proportion to its efficiency in the control of scab.

Treatment 9, which was not greatly different from the standard mercury treatment, gave results similar to treatment 7, except that it did somewhat reduce the total yield, and did not control the runner hyphae on the stems. Treatment 12 was similar in its effects to treatment 9.

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The Treatment of Cut Potato Setts with Zinc Oxide
1. Condition of the Setts, Growth, and Yield

By J. G. Bald, M.Agr.Sc., Ph.D.

Addendum

On page 102 of this article, which appeared in the last issue of the *Journal*, the following caption should have been inserted below Fig. 3:—

> FIG. 3.—Comparison of leaf area, yield, and condition of the setts after allowance has been made for the effects of unequal emergence on leaf area and yield. Mean values for 13 treatments are shown as deviations above or below the regression curves for (*a*) leaf area, January 23, on numbers of plants emerged by November 21 (Table 4B), (*b*) yield on numbers of plants emerged by January 21 (Table 7), and (*c*) sett ratings from Table 1.

The Manna of *Myoporum platycarpum* R.Br. as a Possible Commercial Source of Mannitol

By H. H. Hatt, B.Sc., Ph D., and W. E. Hillis, A.G.Inst.Tech.

Summary.

M. platycarpum under conditions which still remain obscure, yields an exudate in which mannitol forms 60 to 80 per cent. of the total solids. This is not a unique phenomenon, similar exudates having occasionally been observed with other species, particularly the olive tree.

The rates of production of exudate or manna have been measured throughout the year for a few trees. The total solids of the exudate vary from 13 to 26 per cent.; the manna to which it dries usually contains about 90 per cent. of total solids, 70 to 80 per cent. of which is mannitol. In a year a single tree may produce 11 lb. of mannitol. Mannitol was present in the leaves (2·2 per cent.) and the bark (0·6 per cent.) of a healthy tree.

A process for the large-scale isolation of mannitol from this exudate is described. Gum-like materials make crystallization and filtration of mannitol difficult, but these may be removed without the use either of an organic solvent or of charcoal, and mannitol of reagent-grade quality may be obtained by one crystallization in yields of 50 to 60 per cent.

Mannitol is estimated by the method of Badreau. For its detection it is converted to the tribenzylidene derivative, which can be prepared from mixtures of mannitol and glucose containing only 3 per cent. of the former. This method of detection is used in deciding whether certain bacteria can reduce glucose and other sugars. The melting point obtained for tribenzylidene mannitol is considerably higher than that recorded in the literature. Melting occurs with decomposition.

Before an attempt is made to decide whether large-scale preparation of mannitol from this manna is economically possible, there must be found a method of inducing exudation at will and maintaining it over a period of years.

The annual yield of mannitol per tree is comparable with those of rubber and turpentine.

1. Introduction

When Maiden (1) showed in 1892 that the manna obtained from *Myoporum platycarpum* (" sandalwood ") was composed largely of mannitol he was the first to investigate its composition, though, as he stated, it had been described and brought to the notice of scientists many times previously. Later Flückiger (2) published, under the title, " Australian Manna," an able review of the knowledge of mannitol-bearing exudates. There, while admitting that this manna could replace Sicilian manna, he spoke also of the diminishing markets for it as a pharmaceutical, which is possibly one reason why this manna has since received little attention. It is true that, at the same time, he pointed to the remarkable fact that several other trees yielded exudates of high mannitol content, and of apparently closely similar composition, and recommended the determination of the conditions under which these are formed as a worthwhile task for biologists. However, his recommendations seem to have evoked no response.

Since that time mannitol has become an important chemical of commerce. It therefore seemed desirable to examine the value of *Myoporum* manna as a source of mannitol and this paper describes a brief investigation of some problems associated with the isolation of mannitol from the manna.

Flückiger and Maiden both knew that the tree was to be found in the semi-arid regions of all States other than Queensland and Tasmania and described the typical appearance of the manna, which is at first white or pinkish white, but with time becomes reddish or dark brown. They seem not to have examined the liquid exudate, from which the manna is formed by evaporation, and the mannitol content of the manna reported by Maiden is very high (89·65 per cent.). This figure was obtained by difference and is much higher than those we have obtained by direct determination.

The cause of the exudation is still unknown. In investigations which proceeded in collaboration with ours, Fisher (3) characterized two bacteria which are usually to be found in the exudate, but apparently are not the cause of its formation. Exudation can break out on a tree of any age and at any position: on the smallest twig or a few feet from the ground on a trunk 6 feet in girth. The condition is certainly one of disorder and at the seat of exudation the phloem is transformed to a yellow pulp. The appearance of exudation on small branches is rapidly followed by their death; on the larger branches and trunk exudation may cease, leaving large wounds from which the bark lifts exposing the blackened wood beneath. Trabut (4) describes the appearance of an olive tree producing exudate containing mannitol in words which could be applied equally well to *M. platycarpum*.

2. Yields and Compositions of Exudates and Mannas

Investigation of afflicted trees has been limited to a few localities in north-western Victoria, principally at Red Cliffs. No careful survey has been made of the distribution of afflicted trees, but it was apparent during short visits made to the district that the proportion of such trees varies widely. Around Red Cliffs and especially in well-watered positions a third of the trees in a group of 50 might be giving exudate. In the Yarrara State Forest, in one visit, only six afflicted trees were noticed among 300 examined. Around Lake Culluleraine and in the Werrimull district the proportion was even lower.

The trees chosen from which to record the yields of manna and exudate were of fair size and exuding from the trunk. In this position we expected the exudation to continue for some time and it was on the trunk that it was hoped later to induce exudation from sound trees. The trees when selected were producing copious amounts of exudate, but it was not known for how long they had done so and several ceased to exude shortly afterwards. Our resources only permitted us to follow a few trees with the necessary complete analyses of the exudate. Nevertheless, the slender record suffices to show the order of the annual yield from a single tree. The exudate was collected by inserting spouts just below the sites of exudation and attaching cups. The yield was measured daily and in Table 1 the monthly figures are given.

During the summer months the exudate solidifies almost as soon as it emerges (see Tree I. in Table 1) and throughout the greater part of the year, if not collected, most of the exudate solidifies on the tree, producing large lumps and stalactites weighing up to 1 lb. For example, in April, 1944, from a group of seven small trees of girths from 12 to 20 inches, 33 lb. of manna was collected.

TABLE 1.—MONTHLY YIELDS OF EXUDATE AND MANNITOL FROM AFFLICTED TREES.

Month.	Tree I. (Girth 40 in.).		Tree II. (Girth 20 in.).		Tree III. (Girth 18 in.).		Tree IV. (Girth 18 in.).		Group of Seven Trees.	
	Yield.	Mannitol Content.	Yield.	Mannitol Content.	Yield.	Mannitol Content.	Yield.	Mannitol Content.	Yield.	Mannitol Content.
	l.	g.	l.	g.	l.	g.	l.	g.	l.	g.
	(1942)						(1944)			
Sept. ...	7·4	808	..	..	..	..	1·2	171	..	..
Oct. ...	5·7	608	..	..	..	..	1·3	..	..	..
Nov. ...	6·8	811	..	..	..	..	1·2	..	..	..
Dec. ...	3·6	437	2·6	445	..	..	1·2	..	..	..
	(1943)									
Jan. ...	0·5	76	..	..	..	..	..	..	..	..
Feb. ...	0·33*	179	1·1	298	..	..	3·2	585	..	..
Mar. ...	1·11*	448	†	..	..	..	2·2	335	..	..
Apr. ...	0·64*	268	..	..	..	..	0·5	..	..	..
May ...	2·2	278	..	..	..	..	†	..	..	..
					(1944)				(1944)	
June ...	2·8	464	..	..	2·2	234	..	..	8·5	1830
July ...	1·8	190	..	..	2·3	235	.	..	..	..
Aug. ...	2·9	279	..	..	2·1	303	..	..	7·7	1790
Sept. ...	5·8	554	..	..	‡	..	..	..	8·2	1905
Oct. ...	7·2	780	..	..	..	..	..	..	5·4	1625
Nov. ...	6·3	697	..	..	..	..	..	..	7·5	2190
Dec. ...	4·6	981	..	..	..	..	..	..	..	..
	†									

* The exudate concentrates and solidifies during the summer months. These are weights of manna in kg.
† Ceased to exude.
‡ Blown down.

The detailed analyses of exudates given in Table 2 suggest that the considerable variation in the solid content is caused by varying amounts of evaporation depending on the ambient temperature and humidity and the rate at which the exudate issues. This view is supported by the much narrower variation in the percentage of mannitol in the solids, for this rarely exceeds 80 or falls below 60 per cent. Several factors operating after the exudate leaves the wound can explain much of this variation. Exudates are infected to varying degrees with yeasts, and the mannitol will form a greater proportion of the total solids, the greater the extent to which the sugars of the exudate have undergone fermentation. It seems probable

therefore that the composition of the solids in the exudate does not vary greatly from tree to tree or with the season. It might be expected that on a basis of total solids, the proportion of mannitol in manna would be greater than in the exudate, for the mannitol should crystallize before the sugars which might be lost in any liquid flowing away. Fractionation of this kind appears not to be marked. One very clean and crystalline manna from Red Cliffs contained 93·8 per cent. of solids, of which 81·4 per cent. was mannitol and 1 per cent. ash. The mannitol content of this sample was unusually high. Two samples of manna, one a clean 14 lb. sample from Yarrara, the other a single lump from near Lake Cululeraine, contained respectively 89·4 and 90·9 per cent. of solids of which 78 and 73·5 per cent. was mannitol. These mannas came from districts 40 miles from where the material of Table 2 was obtained and serve to confirm the relative constancy of the mannitol content.

TABLE 2.—COMPOSITION OF EXUDATES AND MANNAS.

Tree	Month	Specific Gravity	pH	Total Solids	Mannitol Content	Mannitol in Total Solids	Ash	Total Sugars	Alcohol
		D_4^{20}		%	%	%	%	%	%v/v
Tree I.	(1942) Sept.	1·058	4·1	15·8	10·4	65·4	0·60	1·40	4·8
	Oct.	1·057	4·1	14·9	10·2	68·7	0·64	1·59	3·5
	Nov.	1·060	4·0	16·1	11·3	70·2	0·73	2·44	3·2
	Dec.	1·067	3·9	15·6	11·5	74·1	0·76	1·89	1·4
	(1943) Jan.	1·097	4·4	19·3	13·9	71·9	1·75	..	3·5
	Feb.	Solid	..	86·7	53·8	62·1	10·9	..	..
	Mar.	,,	..	58·0	40·5	69·9	4·59	..	..
	Apr.	,,	..	59·7	42·0	70·3	5·80	..	.
	May	1·077	5·3	17·8	11·9	66·6	1·52	..	..
	June	1·075	5·3	19·3	14·3	74·3	1·31	..	..
	July	1·055	4	13·9	10·1	73·7	0·96	.	..
	Aug.	1·058	5	12·8	9·0	70·5	1·09	..	..
	Sept.	1·055	5	12·7	8·9	70·4	1·04	..	..
	Oct.	1·056	5	13·2	10·3	78·5	0·71	..	..
	Nov.	1·055	5	13·4	10·4	78·5	0·78	..	..
	Dec.	1·121	5	23·6	19·1	81·4	2·12	..	..
Tree II.	(1942) Dec.	1·101	4·3	23·9	15·9	66·2	0·92	2·26	3·3
	(1943) Feb.	1·150	4·9	32·4	23·0	70 9	..	..	2·0
Tree III.	(1944) June	1·057	4	16·4	10·0	61·4	0·55	..	..
	July	1·054	4	16·3	9·7	59·3	0·42	..	..
	Aug.	1·085	5	20·6	13·2	63·6	1·05	..	..
Tree IV.	(1944) Sept.	1·079	5	20·8	13·4	64·2	1·34	..	..
	(1945) Feb.	1·095	4	23·8	16·6	70·0	1·10	..	..
	Mar.	1·078	4	21·2	14·2	66·8	0·78	..	..

Some more complete analyses of the exudate were made as a guide in developing a method for the isolation of mannitol. Methods of analysis are given in a subsequent part of this paper. The following is a typical composition:—

Percentage Composition of Total Solids.
(Exudate for December, 1942, of Table 2.)

Mannitol	74·1
Reducing sugars (as glucose)	8·0
Polysaccharides	4·23
Protein (nitrogen $\times$ 6·25)	1·44
Volatile acids (as acetic acid)	1·48
Non-volatile acids (as lactic acid)	2·96
Total ash	4·83
(Alkaline ash as K_2CO_3)	(3·06)
	97·0

Although the exudate cannot be obtained by incision, there is evidence that mannitol production is not a result of a pathological condition in *Myoporum platycarpum*. McDowell (5) found 0·20 per cent. in the green leaves and 0·25 per cent. in the undried wood of the New Zealand species *Myoporum laetum,* and Albert (6) isolated 0·14 per cent. from the dried leaves of *Myoporum deserti.* Much larger quantities are present in the leaves and bark of *M. platycarpum.* We isolated 2·2 per cent. of pure mannitol from the dried leaves and 0·6 per cent. from the bark of healthy trees. While the seasonal variation of the mannitol content was not examined, this high value suggests that the mannitol in the exudate could well be produced by the normal metabolism of the tree.

3. Isolation of the Mannitol

Until recently mannitol of commerce had been prepared exclusively from Sicilian manna and it might have been expected that the methods of isolation used would apply to *Myoporum* manna. A satisfactory account of the methods employed is difficult to find and the literature available indicated that the processes used were not very efficient. Ruspini in 1848 (7) first developed a process that avoided the use of alcohol and his seems to have been the one commonly used. The manna was dissolved in a little hot water and the solution, after clarifying with egg-white, was filtered, cooled, and the crystalline magma pressed and recrystallized from water after decolourizing with charcoal. The proportion of mannitol recovered is not recorded, but Scarlata (8), in 1921, commented adversely upon the method and sought improvement by introducing a cold-water wash of the manna to remove molasses before crystallizing the mannitol. He too gives no indication of the proportion of mannitol isolated.

Like these authors, we set out to develop a method of isolation which avoided the use of alcohol or other organic solvent. The exudate, which is at first an almost colourless, slowly fermenting liquid, with an odour charactertistic of mixed alcoholic and acetic fermentations, darkens with time and deposits a dark brown, flocculent sediment. Boiling accelerates the darkening and the precipitation, and after five or six hours, if the exudate is boiled for another hour with 2 or 3 per cent. of charcoal and filtered, the colourless liquid becomes only pale brown by further boiling and yields no precipitate.

This liquid can be concentrated under reduced pressure and the mannitol that then crystallizes on cooling is white. Such a method of isolation, while suitable for a few grammes of mannitol, failed for larger quantities, because the filters were quickly blocked by the small amounts of gum still present. Many other methods were tried, including the use of larger quantities of charcoal and of other adsorbents, and the processes of single and double carbonatation as practised in sugar refining. None were suitable; they either needed too much costly adsorbent or involved filtration processes that were too slow.

Except that the exudate contains mannitol whereas sugar cane juice contains sucrose, these two liquors resemble one another closely and the method of isolation we have developed for mannitol is a modification of one used in sugar refining. However, filtration was always difficult and the gums could only be removed in satisfactory manner on a Sharples super-centrifuge. The procedure used for the removal of gums and precipitable matter involved four stages, at the end of each of which the liquors were centrifuged. In the first, coarse material was removed on a sieve and the finely suspended matter was afterwards separated in the centrifuge. The three subsequent stages consisted of (i) boiling, (ii) liming to an initial pH 9 followed by boiling, and (iii) the addition of trisodium phosphate to remove excess of lime. The optimum conditions for each operation were discovered by trial. It was found that less gums and impurities were removed if centrifuging after any one of these operations was omitted.

When the liquors were boiled, highly coloured gelatinous material continued to separate for about ten hours, but as after four hours the precipitate did not cohere well and was difficult to remove in the centrifuge, boiling was limited to this period. About 60 per cent. of the total amount precipitable was removed at the end of four hours.

In the clarification with lime, we examined the dependence of the amount of organic matter precipitated upon the final pH of the liquid and the time of boiling. The amount increased threefold while the pH rose from $6 \cdot 0$ to $7 \cdot 3$ and began to decrease again at pH 8. Boiling for one or two hours was sufficient and the amount of organic matter precipitated decreased appreciably if it was continued for three hours. In liming, the clarification process known in the sugar industry as Fractional Liming and Double Heating (9) was used and slightly increased the amount of organic insoluble matter removed. The liquid was heated to boiling, sufficient milk of lime added to raise the pH to $8 \cdot 0$, and after boiling for an hour, during which the pH fell to $7 \cdot 2$, additional lime was added to raise the pH to $9 \cdot 5$, which fell in a further one hour's boiling to $8 \cdot 0$.

Precipitation of the excess lime with trisodium phosphate gave the best results when carried out at room temperatures.

The organic materials precipitated in these four stages differ in composition. Most of the nitrogenous matter appears in the first precipitate, and the second is richest in carbohydrates. Table 3 collects the analytical data for these precipitates.

When once the gums had been removed, concentration of the aqueous liquors, crystallization of the mannitol, and subsequent filtration proceeded without difficulty. Concentration to a syrup containing 15–20 per cent. of water gave the best yield of mannitol and if this syrup was cooled slowly and stirred continuously the

TABLE 3.—CLARIFICATION OF EXUDATE.

Clarification Process.	Total Solids Precipitated.	Composition of Precipitate (" Gums ").			
		Ash.	Protein (6·25 x N%).	Reducing Sugars.	Hydrolysable Sugars.
	%	%	%	%	%
Centrifuging cold liquors ..	0·4	17	22	2·8	5
Centrifuging boiled liquors	0·1	25	8	4·8	17
Liming	0·9	33	0·5	0·9	11
Lime removal with phosphate	2·0	38	..	..	..

crystals were of uniform size. Although the liquors were dark brown, a good grade of mannitol was obtained without decolourizing. The crystals, after separation from the molasses, were washed twice with small amounts of a saturated aqueous solution of mannitol, and were then white and practically pure.

(i) *Preparation of Mannitol on a Laboratory Scale.*

The manna used was of good quality from the Yarrara district. It contained 69·8 per cent. of mannitol which formed 78·1 per cent. of its total solids. 2·8 kg. were dissolved in 9·5 litres of water, passed through a 10 mesh sieve to remove debris (twigs, leaves, and bark) and then through a laboratory Sharples super-centrifuge running at 20,000 r.p.m. Details of the run are given in the flowsheet.

(a) *Clarification.*

Manna (2·8 kg.)   Water (9·5 kg.)

```
Manna (2·8 kg.)                    Water (9·5 kg.)
    |_____|
                     |
                     | Screened ————————>Debris (1·8 per cent.
                     V                      of manna)
               Liquor A.
                     |
                     | Centrifuge ————————>Gums  A.—Dry  wt.
                     V                      1·5  per  cent.  on
                     |                      manna.   Mannitol
               Liquor B.                    content 17 per cent.
                     |
                     | Boil 4 hours, cen—>Gums  B.—Dry  wt.
                     V    trifuge          0·07 per  cent.  on
                     |                      manna.   Mannitol
               Liquor C.                    content 17 per cent.
                     |
Lime (CaO) (1·1 per cent. on—>—| pH 9·1, boil 1 hr.
   manna) as milk of lime, S.G. V  pH 7·0
   1·115
Lime (0·85 per cent.) as milk of—>—| pH 9·5, boil 1 hr.
   lime                             pH 8·0
                     |
                     | Centrifuge ————————>Gums  C.—Dry  wt.
                     |                      4·2  per  cent.  on
                     |                      manna.   Mannitol
                     |                      content 21 per cent.
               Liquor D.
                     |
Na₃PO₄·12H₂O (6·13 per cent.—>—| Mix and centrifuge—>Gums  D.—Dry  wt.
   on manna) added as 10 per     V                    4·8  per  cent.  on
   cent. solution                |                     manna.   Mannitol
                           Liquor E.                   content 20 per cent.
```

Liquor E was of pH 7·3, S.G. 1·074 and contained 17·8 per cent. total solids and 14·6 per cent. of mannitol; 82 per cent. of the total solids were mannitol. Larger proportions of gums were removed in this run than are recorded in Table 3, because they were not freed of the mother liquor which is rich in mannitol and other soluble substances. In commercial practice washing and recentrifuging the gums would not be feasible. Those in Table 3 were washed to discover the nature of the insoluble substances.

(b) *Crystallization.*

Liquor E. (11·5 litres containing 1·8 kg. mannitol)

↓ Concentrate at 12 cm. ＞ Distillate rejected
pressure

Syrup (2·78 kg., water content 21 per cent.)

Cool for 24 hours ＞ 1380 g. Molasses (S.G.
↓ with stirring, 1·283)
filter on cloth in
basket centrifuge

Residues

Wash with 500 ml. of saturated— ＞ — | —————————— ＞ —Wash liquor, 470 ml.
mannitol solution, mannitol
content 100 g.

Wash with 400 ml. of solution,— ＞ — | —————————— ＞ —Wash liquor, 360 ml.
mannitol content 80 g.

↓

Mannitol
Dried at 100°C., yield 1310 g., m.p. 165–166°C.

This represents a gross yield of 67 per cent. When the mannitol used in the wash liquors is deducted the net yield is 58 per cent. The molasses contained 30 per cent. of mannitol or a little over 21 per cent. of that present in the manna. Altogether 97 per cent. of the mannitol was accounted for and this figure lies within the total experimental errors of the various mannitol estimations.

(ii) *Isolation of Mannitol on a Pilot Plant Scale.*

The material available was sufficient for two larger runs. A good grade manna was used in the first, which suffered from the usual minor troubles and losses. In the second, reported here, a mixture of mannas was used, some of very low grade and containing much extraneous matter.

No specially designed equipment was used in this work. A stainless steel tank served as containing vessel for the clarifying processes and was provided with a closed steam coil and a mechanical stirrer. For concentration of the clarified liquors we used a converted 30-gallon iron digester, fitted with a 2-in. vapour outlet pipe, a water-cooled condenser, and a receiver for the distillate. The Sharples super-centrifuge was run at 30,000 r.p.m. and the liquor was fed in at rates varying from 25 to 35 gallons per hour and at a temperature of 35°C.

For the second isolation, 480 lb. of manna was used. It was dissolved in 80 gallons of boiling water and freed of debris (41 lb.) by passage through a 14-mesh wire gauze in a basket centrifuge. The liquors obtained (109 gallons) had a total solids content of 30·4 per cent. of which 73·4 per cent. was mannitol. The manna therefore contained 276 lb. (57·5 per cent.) of mannitol.

Clarification which followed closely the laboratory procedure gave finally 122 gallons of liquor E which were concentrated at a pressure of 5 inches to a water content of 20 per cent. The syrup was discharged into drums and there cooled and stirred for three hours. After 24 hours the crystals were collected on 100-mesh gauge in a basket centrifuge, removed, stirred with a saturated aqueous mannitol solution, and recentrifuged. This washing process was repeated. The total saturated mannitol solutions used for washing contained 34 lb. of mannitol. The washed mannitol was dried at 100°C. at 2 inches pressure. It melted at 165·5 to 166·5°C., weighed 173 lb., and represented a gross yield of 63 per cent. (net yield, 139 lb. or 50 per cent.). Concentration of 3 litres of liquor E on a laboratory scale resulted in a gross yield of 61 per cent. and a net yield of 50 per cent. The lower grade of manna used in this pilot plant run is therefore responsible for the lower yield rather than unrecognized factors introduced in increasing the scale of operations.

Tables 4, 5, and 6 give the compositions of the molasses and of the various gums and liquors and show the distribution of the mannitol at the completion of the experiment. Some 98 per cent. of the mannitol is accounted for with 34·5 per cent. in the molasses.

TABLE 4.—ANALYSIS OF PROCESSING LIQUORS.

Liquor.			S.G. $\frac{20}{4}$	pH.	Calcium as $CaCO_3$.	Total Solids.	Mannitol.
					%	%	%
A	..	..	1·132	..	..	30·4	22·3
C	..	..	1·120	..	..	29·2	21·7
D	..	..	1·120	7·5	0·53	28·8	21·4
E	..	..	1·110	7·0	..	25·7	19·5

TABLE 5.—ANALYSIS OF GUMS.

Gum.			Water Content.	Dry Weight.	Mannitol Content.	Mannitol Content
			%	lb.	%	lb.
Debris	..	..	23	41	..	..
A	..	..	44	11	22	2·4
B	..	..	50	2·5	39	1·0
C	..	..	65	6	33	2·0
D	..	..	37	22	29	6·3
					Total ..	11·7

TABLE 6.—ANALYSIS OF FILTRATES.

Filtrate.	S.G. $\frac{20}{4}$	Weight.	Total Solids.	Mannitol.	Mannitol Present.
		lb.	%	%	lb.
Molasses	1·295	303	59·4	31·5	95·4
Wash Liquor 1 ..	1·172	80	26·9	21·2	17·0
Wash Liquor 2 ..	1·081	96	20·1	19·3	18·5
				Total ..	130·9

The melting point and almost white appearance of the mannitol isolated in this run attest its high purity. It fulfils the requirements for reagent-grade mannitol (10). The products from laboratory and pilot plant runs differ but slightly in composition as is shown in the comparison of analytical data in Table 7. The chloride ion and iron contents are reported because they are of importance in deciding the value of the mannitol for use in electrolytic condensers. As might be expected the mannitol from the pilot plant has a slightly higher content of iron.

TABLE 7.—ANALYTICAL DATA FOR MANNITOL ISOLATED.

Run.	Melting Point	Moisture	Insoluble in Water.	Total Ash.	Ferric Iron.	Chloride (as NaCl).	Sugars.
		%	%	%	%	%	
Laboratory Scale	165–166°C.	0·11	0·17	0·15	0 001	0·017	Absent
Pilot Plant ..	165·5–166·5°C.	0·07	0·03	0·05	0·005	0·016	Absent

4. Discussion

(i) *Isolation.*

A simple process has been devised by which 50 per cent. of the mannitol of an exudate, or manna, can be isolated in a state of purity. Considering how this yield could be increased, first it can be noticed that 10 to 12 per cent. of the mannitol remains in the wash liquors. These would normally be added to the next batch of manna and might be expected to raise the net yield to about 60 per cent. To improve this figure appreciably, further mannitol must be recovered from the molasses, which contains the greater portion of the remaining 40 per cent. On the laboratory scale an additional 8 per cent. of mannitol was recovered if the molasses was mixed with an equal volume of 75 per cent. aqueous alcohol, but so far no appreciable quantities of mannitol have been obtained from the molasses by processes which avoid the use of an organic solvent.

When the clarified liquors were concentrated under reduced pressure, it was noticeable that any superheating caused caramelization and reduced the yields of mannitol. Presumably caramelization

was restricted to the sugars present, for mannitol determinations on the molasses agreed with the amounts calculated to be there and were taken to show the absence of any appreciable formation of anhydro-mannitols. The process of isolation we have described was devised especially for the exudate. If manna should be the material processed, the removal of a large proportion of the sugars and hydroxy acids by a preliminary treatment might lessen the amount of molasses and increase the yield of mannitol. This could be done by mixing the manna with a small quantity of water and pressing, as Scarlata (8) proposed to treat Sicilian manna. Whether it would be economical to convert exudate to manna for submission to this process would depend on the resulting increase in yield. Further work in these directions seems unwarranted as long as the possibility of inducing exudation at will remains remote.

(ii) *A Comparison of Yields of Organic Compounds Prepared from Various Exudates.*

The present knowledge of the yields of manna make it certain that *Myoporum* manna is potentially a far superior source of mannitol than the manna of the Sicilian manna ash (*Fraxinus ornus*). According to Molinari (11) the Sicilian manna harvest continued throughout August and September and involved daily incision of the trees. The trees were planted about 1,800 to the acre and these in a season gave about 40 kg. of manna. He cites the mannitol content of the manna as 32-42 per cent., so that the yield of mannitol was about 10 g. per tree. Other more general accounts omit figures for the yields, but in the main confirm the laborious methods and low yields Molinari describes. Nevertheless, in 1918 over 200 tons of manna were exported from Sicily (11). Since then this manna has been superseded as a source of mannitol by glucose, from which the mannitol is prepared by electrolytic reduction.

As shown in Table 1, Tree I. produced at the rate of 11 lb. (5 kg.) of mannitol per year. This yield is 500 times that from the Sicilian manna ash and, in fact, is comparable with yields of rubber and turpentine, which also are produced as exudates and are the raw materials for large organic chemical industries. According to Dorman (12) the annual yield of turpentine per pine tree in the U.S.A. averages about 8·3 lb. with single trees giving as much as 12 lb. Memmler (13) quotes the average yield of rubber per tree as 20 g. daily or 2 kg. in a tapping season with single trees producing up to 200 g. daily or 12 kg. in a season. Both pine and rubber trees have long productive lives, whereas that of *M. platycarpum* is unknown. However, these comparisons do show that the yield is sufficiently large to consider it as a probable economic source of mannitol.

The yield of mannitol can be viewed in another setting. Seth Smith (14) has shown that some land, when devoted to sheep grazing, returns no more than one shilling per acre per year, whereas like land with its natural planting of *Eucalyptus dives* returns annually over 12 shillings per acre when harvested for oil. *Myoporum platycarpum* grows in regions of low rainfall and its value for the production of mannitol and timber, and as a protection against erosion merits consideration.

(iii) Alternative Methods for the Production of Mannitol.

The production of mannitol by the electrolytic reduction of glucose has proved commercially the most successful of the many methods proposed (15). When the Atlas Powder Company commenced large-scale manufacture by this method in 1937 the cost of mannitol in the U.S.A. was 4 dollars per pound. Pure mannitol is now marketed at 85 cents per pound and a commercial grade at 40 cents. Mannitol from manna would have to be produced at a competitive price, and in Australia this probably would be not more than 4 or 5 shillings per pound for the pure compound.

(iv) Cause of the Exudation.

Fisher (3) has already discussed this subject and we intend only to emphasize that the formation of an exudate rich in mannitol is not peculiar to *Myoporum platycarpum*. Flückiger when he reviewed the knowledge of mannas mentioned two other mannas which closely resembled Australian and Sicilian mannas. Since then several others have been described* and in the introduction, reference has already been made to the close similarity in the appearance of exuding olive and *M. platycarpum* trees. It seems clear that the formation of such an exudate is not restricted to any one species, but is a phenomenon widely, though sparsely distributed among plants. According to Zanda (16), whether the Sicilian ash yields as exudate on incision even depends on its location; for example, trees grown in northern Italy do not exude when incised. Perhaps 50 years afterwards is an opportune time to repeat Flückiger's suggestion that this phenomenon is one worthy of the attention of plant physiologists.

5. Experimental

(i) Mannitol Content of the Leaves and Bark of M.platycarpum.

One kilogramme of leaves (collected in August from healthy trees were minced and boiled three times with 3 litres of water, the extract being squeezed from the leaves through a silk bag. Preliminary experiments had shown that steam distillation removed very little essential oil and that a fourth extraction with boiling water contained little if any mannitol. The aqueous liquors were concentrated at 3 to 4 cm. pressure on a water-bath at 60-65°C. and when about 100 ml. in volume, the extract was mixed with two volumes of 10 to 20 mesh quartz and dried in vacuum at 64°C. The dry material was extracted with absolute alcohol at its boiling point, the alcohol

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* Plants reported to give mannas of high mannitol content are:—
  Fraxinus ornus L. and *Fraxinus rotundifolia*.
  Myoporum platycarpum R.Br.
  Platanus orientalis: Jandrier, M.—*C.R. Acad. Sci. Paris* 117: 498 (1893).
  Olea Europea L. (the olive): Trabut.—*Ibid.* 132: 225 (1901).
  Andropogon annulatus Forsk.: Baker and Smith.—*Apoth. Ztg.* 12: 326 (1897).
  Alhagi camelorum Fisch. (Tarandjabine manna): Hudson, C. S., and Sherwood, S. F.—*J. Amer. Chem. Soc.* 40: 1459 (1918).
  Gardenia turgida: Forster, M. O., and Rao, K. A. N.—*J. Chem. Soc.* 125: 2176 (1925).
  Cape Verde Manna (tree unidentified): Berthelot, D.—*Ann. Chim.* (3) 46: 83 (1856).

evaporated and the product extracted with acetone. Evaporation of the acetone left a light brown crystalline material which after crystallization from alcohol melted at 162·5-164·5°C. The hexa-acetate melted at 123·5°C. and did not depress the melting point of an authentic specimen. The bark of the tree was ground to 40 mesh and extracted in like manner. The results are summarized in Table 8.

TABLE 8.

Material Used.	Moisture Content	Yield of Mannitol (Percentage Dry Basis).	Melting Point of Mannitol.
	%		°C.
1000 g. Leaves	52·7	2·26	162·5-164·5
850 g. ,,	52 7	2·20	162 -164·5
200 g. Bark	11·6	0 59	164 -165·5
300 g. ,,	11 6	0·62	166

(ii) *Analytical Methods.*

(a) *The determination of mannitol in manna and in exudate.*

The methods for determining mannitol in solution which investigations have shown to be least affected by the presence of other substances, are those based upon the enhanced optical rotation which results when certain inorganic acids or their salts are added. Borax has been most used, but it requires the previous complete elimination of sugars from the solution. Sugars formed 8 to 15 per cent. of the total solid content of the exudates we received and it was not found possible to eliminate them by fermentation. The substitution of ammonium acid molybdate for borax was recommended by Frèrejacque (17) and its greater enhancement of optical rotation is an advantage, but since Salani (18) found the method applicable only when small amounts of sugars were present, we chose the method of Badreau in which sodium arsenite is added (19). Although less accurate, for it cannot be used with a mannitol concentration greater than 1·7 per cent. which, in a 4 dcm. tube, gives an enhanced rotation of only 1·26°, it has the advantage that relatively large proportions of sugars do not interfere. We have found glucose, sucrose, sodium acetate, and meta and ortho-phosphoric acids without influence when present in amounts up to 10 per cent. of the weight of mannitol. Larger quantities of sugars (concentrates of 20 per cent.) interfere, for on addition of the sodium arsenite solution the observed rotations differ from the calculated values as follows:—

Sucrose	.. .. ..	—0·43°
Glucose	.. .. ..	—0·51°
Xylose	.. .. ..	—1·46°
Sorbose	.. .. ..	+1·12°
Lactose (10 per cent. solution)		+0·89°

. In the procedure adopted for the determination of mannitol, a measured volume (50 to 100 ml.) of the exudate, or of a 20 per cent. solution of manna, was diluted to 200 ml., 2–5 g. of charcoal added, and the whole boiled gently for three to four hours to remove alcohol and acetic acid and to coagulate gums. Sodium hydroxide was then added until the pH was 8 or 9 to precipitate phosphates, after which the liquors were filtered, the charcoal washed repeatedly with boiling water, and the filtrates made up to 1 litre. Two 20-ml. aliquots of this liquid were diluted to 50 ml., the one with water and the other with Badreau's sodium arsenite solution and their rotations determined at approximately 20°C. To obtain the maximum accuracy, determinations were made at several dilutions with one reading close to the maximum permissible rotation. In some determinations, prior to the use of charcoal, a lead acetate clarification was introduced to remove hydroxy acids, which Badreau stated interfered with the determination. Actually no change in the mannitol determinations resulted from this refinement so that it was used only occasionally as a check.

(b) The determination of mannitol in cultures.

In the peptone-water and Czapek-Dox cultures of the bacteria isolated by Fisher (loc. cit.) the estimation of mannitol was performed according to the method of Badreau after a clarification with lead acetate. No positive results were obtained.

(c) Reducing sugars.

The presence of glucose in the exudate was established by the dextro-rotation of the liquid and by the preparation from it of glucosazone. This osazone was formed in a reaction time of five to ten minutes; it melted at 210°C. and possessed the characteristic glucosazone habit. Reducing sugars were estimated by the modified method of Munson and Walker, Official Method, A.O.A.C., 1940, 500, which involves preliminary clarification with lead acetate and removal of lead as oxalate.

(d) Polysaccharides.

These sugars were hydrolysed by heating for fifteen minutes with 20·9 per cent. hydrochloric acid and the resulting reducing sugars estimated according to Official Method, A.O.A.C., 1940, 494.

(e) Volatile organic acids.

The method of Fred, Peterson, and Davenport (20) was used for the estimation of volatile acids in the exudate, with the modification that to avoid distillation of any hydrochloric acid liberated by the phosphoric acid, a small quantity of silver sulphate was added. The presence of acetic acid in this fraction was established by preparation of the phenylphenacyl derivative.

(f) Non-volatile organic acids.

The method of Fred, Peterson, and Davenport (20) for the estimation of lactic acid was used. The presence of lactic acid in this fraction was established by preparation of the phenylphenacyl derivative. '' The non-volatile acids of the exudate were tested for succinic acid according to the method of these authors. It was absent.

(g) Alcohol.

Alcohol was estimated by distillation and determination of specific gravity. It was identified through the preparation of its 3 :5- dinitrobenzoate.

(h) Total solids.

The solid from the exudate lost weight continuously at 100-105°C. and the weight was recorded after drying for 96 hours.

(i) Ash.

The total solids were dried overnight at 150-160°C. and afterwards brought slowly to 500°C. and ashed.

(iii) *The Precipitation and Determination of Gums.*

The optimum conditions for clarification have already been described as part of the method used for the isolation of mannitol. These were determined from a series of small-scale experiments upon 100 g. samples of exudate, from which the amount and nature of the gums precipitated were studied while the more important factors of influence were varied. It is not necessary to report these experiments in detail. Separation of the gums in fractional manner was adopted since it was considered likely that some precipitates would be peptized with a change in conditions, and in fact in practice the largest quantities of gums were collected when each fraction of gum was centrifuged separately. In the sugar industry it is known that 70 per cent. of the non-sugar substances eliminated by lime defecation can be flocculated by heat alone. Moreover we found removal of these thermally unstable dispersed materials did diminish slightly the amount of lime required.

In these experiments the gums were separated by batch centrifuging for one hour at 500 times gravity. The gums adhered firmly to the glass cups and their quantitative removal presented much difficulty, until thin cellulose acetate sleeves were made for the cups. These were made in the usual manner by running in a solution of the resin (Hercules Powder L.M.I.) in ethyl acetate. The sleeves were about 0·002 inches in thickness and could be detached and dried to constant weight at 105°C. (two hours). For our purpose the amount of ash they contained was negligible (0·1 per cent.). The gums could thus be centrifuged into the sleeves, the liquors siphoned off, the gums stirred with 25 ml. of water, re-centrifuged, and, after the wash liquors had been removed, dried to constant weight.

Gums A and B contained much iron, but very little calcium or phosphate.

For defecation the lime used was that supplied to the Colonial Sugar Refinery, Yarraville, for carbonatation. Our sample contained 78·8 per cent. of active lime. Following Davies and Yearwood (21) we used the thick milk of lime of 15° Bé (180 g. lime per litre) which they reported to give greater elimination of non-sugars, a decreased turbidity and a lower residual ash. Gums C contained considerable quantities of iron and phosphate.

It is the practice in sugar refining to remove excess calcium left after defecation by the addition of trisodium phosphate. The calcium was estimated volumetrically according to Lingane (22), except that it was necessary first to remove colouring matters by evaporating the 25 ml. aliquot to dryness with 10 ml. of 12 N HCl, followed by re-solution in 40 ml. of boiling water and neutralization, before precipitation of the calcium oxalate.

Estimations of protein (indirectly as nitrogen), reducing sugars and hydrolysable sugars in the gums followed mainly well-known procedures.

(iv) *The Conversion of Mannitol into Tribenzylidene Mannitol as a Means of Detection in the Presence of Sugars.*

Although the method of Badreau for the determination of mannitol is relatively free from interference by sugars, the low rotations measured introduce uncertainty into determinations of small quantities of mannitol and there was a need to support these determinations by demonstrating the presence of mannitol through the preparation of a derivative. The preparation of tribenzylidene mannitol was selected for trial and the method finally used was a slight modification of that given by Fischer (23). Mannitol (2·5 g.) was shaken with hydrochloric acid of density 1·17 (7·5 ml.) until it had dissolved, when freshly distilled benzaldehyde (5 ml.) was added and the reaction mixture was shaken until it had set to a solid mass. After allowing to stand for 24 hours, ice-cold normal sodium hydroxide (95 ml.) was added to the reaction mixture with agitation, and the liquors subsequently made alkaline with normal sodium carbonate. The product was taken up in chloroform, water added, and the benzaldehyde removed in steam. (Removal of the first aqueous layers before steam distillation suppressed the formation of gums from the sugars. In a simple preparation of tribenzylidene mannitol these aqueous liquors need not be removed.) The residues were again dissolved in chloroform, freed of benzoic acid by washing with sodium carbonate solution and with water, dried, and the solvent removed. The residues weighed 5·5 to 5·6 g. (90 per cent.) and melted at 217-220°C. They were further purified by crystallization from benzene.

Various modified procedures were tried, but the above proved most suitable. The hydrochloric acid used in the preparation could be reduced to 0·1 ml. if 7 ml. of acetic acid and 4 ml. of acetic anhydride were also added, but the yield was then only 70-75 per cent. An 80 per cent. yield was obtained using 0·3 ml. of 60 per cent. perchloric acid alone, which was raised to 90 per cent. when the amount of benzaldehyde was doubled. Using a like excess of benzaldehyde a 90 per cent. conversion of mannitol to its derivative was obtained in the presence of 1 ml. of 90 per cent. (S.G., 1·75) phosphoric acid. When attempts were made to drive the reaction to completion by azeotropic removal of the water with ethyl acetate no tribenzylidene mannitol could be isolated.

Benzalating according to the chosen method, it was found that 0·06 g. of mannitol could be detected in the presence of 40 times

its weight of glucose (2·5 g.) or twenty times its weight of sucrose. The tribenzylidene mannitol first obtained from treatment of such mixtures was very impure, but the amount sufficed for the preparation of a pure specimen by several crystallizations from benzene-petroleum ether. By Badreau's method 2 or 3 parts of mannitol can just be detected in the presence of 100 parts of glucose so that this derivative can be used to detect mannitol at about the same dilution.

Both methods were employed to decide whether mannitol had been produced in Czapek-Dox or peptone-water cultures of the bacteria isolated by Fisher (3) from exuding portions of *Myoporum platycarpum* trees. In the attempted isolations of tribenzylidene mannitol, 100 ml. of the culture was concentrated on a water bath under reduced pressure to a thick syrup, shaken with 39 per cent. hydrochloric acid (15 ml.) until it had dissolved and then for two hours with benzaldehyde (10 ml.). After standing 24 hours the product was worked up in the manner already described. As stated by Fisher, mannitol was not found in the cultures.

(v) The Melting Point of Tribenzylidene Mannito.

Use of this compound as a derivative drew our attention to the wide range of values reported for its melting point. Values from 207°C (Meunier (24)) to 224°C. (Pette (25)) have been assigned, the range 213-219°C. being commonest. We have found that after repeated crystallization either from chloroform or benzene, the melting commences at 224°C. but is not complete until 230-234°C. This behaviour can be explained by thermal decomposition, for tribenzylidene mannitol liquefies completely when kept for three hours at 205°C. or for 7 hours at 195°C., and before these times sublimates of benzoic acid can be seen. However, when the compound is plunged at 224°C. into a bath of rising temperature, it melts at 225-227°C. suggesting that higher melting points result from dimorphism or the interconversion of stereoisomers. No separation into fractions of different properties has so far resulted from fractional crystallization of tribenzylidene mannitol.

Our pure material gave $[\alpha]_D^{20}$, —16·2° in chloroform (C, 8·3 per cent.). Pette reported $[\alpha]_D^{17}$, —16·5° in chloroform (C, 7·019 per cent.) and Patterson and Todd (26), $[\alpha]_D^{25}$, —15·5° in chloroform (C, 1·056 per cent.).

6. Acknowledgments

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Furfural: A Pilot Plant Investigation of Its Production from Australian Raw Materials

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Summary.

A pilot plant investigation of a single-stage digestion process for the production of furfural from oat-hulls and maize-cores is described. The variations in yield obtained over a wide range of operating conditions are given, together with the data necessary for the design of full-scale digestion equipment.

. A critical study of the recovery of furfural from the digestion products by distillation is described and the necessary data are given for the design of column condensers.

The cost of production of furfural in Australia from oat-hulls was found to be approximately 1s. per pound, based on a plant designed for the production of 200 tons of furfural per year.

1. Introduction

The purpose of the work described in this paper was to collect and summarize previously published data on the manufacture of furfural and to design and operate a pilot plant suitable for the production of furfural from Australian agricultural waste materials. Sufficient data were obtained to permit the rational design of a large-scale plant to suit Australian conditions and to permit a preliminary estimation of production costs.

Furfural is produced in aqueous solution by the steam distillation under pressure in the presence of a catalyst, of a wide variety of agricultural wastes such as oat-hulls, maize-cores, and other cellulosic materials and is recovered from the distillate by fractional distillation. The earliest commercial development of this process occurred in the United States of America where attempts (1-7) were made to derive useful products, in particular furfural, from maize-cores and later from oat-hulls (8). A pilot plant investigation by La Forge and Mains (9) showed that the industrial production of furfural was economically feasible, and work on the distillation of furfural solutions by Mains (10, 11, 12) greatly facilitated the development of the commercial production of furfural. The industry developed rapidly after the introduction of a process for refining lubricating oils (13) in which furfural is used as a selective solvent. The design and operation of plants developed during this period has been described by Killeffer (14) and Brownlee (15). In contrast to the development of American processes, European industry has favoured processes in which furfural is produced in conjunction with pulp or alcohol (16, 17, 18). Recently this type of process has been re-investigated in America (19) with a view to the simultaneous production of furfural and alcohol from agricultural wastes.

The main difference between these processes is in the methods employed for digestion. In the American process the pentosan hydrolysis and the dehydration of pentoses to furfural are carried out

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in a single stage using high pressure or superheated steam. The European processes employ a two-stage digestion consisting of a low temperature pentosan hydrolysis followed by the dehydration of the pentoses at elevated temperature after their separation from the cellulosic material. Both types of digestion can be carried out batchwise or continuously.

In selecting a suitable digestion process for pilot plant development, continuous methods were eliminated because of the probable small demand for furfural in Australia, and the use of superheated steam was avoided because of the expected difficulties of agitating and discharging the semi-solid mass from the digester. The process selected for investigation was the single-stage digestion and steam distillation of the pentosan-containing material at elevated pressure in the presence of dilute sulphuric acid. Also, some aspects of a two-stage process were investigated by Wilson*.

Furfural can be obtained from any pentosan-containing material; the most favoured materials for its production are oat-hulls and maize-cores, although such diverse materials as rice-hulls, flax-shives, straw, and wood have been used (20). A survey of the potential raw materials in Australia which took into account their pentosan content and probable costs, led to the choice of oat-hulls as the main raw material for this investigation, because in Australia oats are milled at centres close to existing chemical industries while maize is milled at country centres.

The major use of furfural is as a cheap solvent and particularly as a selective solvent in the refining of lubricating oils, but its chief uses in Australia are as a substitute for formaldehyde in phenol formaldehyde resins and as a solvent in the paint and coatings industries.

For convenience in presentation the description of the work has been divided into the following sections:—

> The Process Study of the Digestion.
> The Pilot Plant Investigation of the Digestion.
> The Recovery of Furfural from the Digestion Product.
> The Cost of Furfural Production.

2. The Process Study

Before designing the pilot plant it was necessary to have some knowledge of the relative importance of the variables of the digestion process, and, in particular, it was necessary to know the limits within which the pilot plant would be required to operate.

These variables were as follows:—

> 1. Pressure and temperature of operation.
> 2. Catalyst concentration.
> 3. Period of digestion.
> 4. Steam throughput.
> 5. Liquid/solid ratio.
> 6. Type and degree of agitation.

Some of this information was available in the literature, but to determine the remainder and to verify published results for Australian raw materials a study was undertaken in equipment available at the time.

(i) The process equipment.

The digester used for the process study was a copper-lined, mild steel, steam-jacketted unit designed to operate at pressures up to 200 lb./sq. in. The digester, which was 24 inches in diameter by 24 inches deep, was fitted with a slow-speed paddle-type stirrer, a filling opening, vapour outlet line, waste discharge valve, and all necessary pressure gauges and safety valves. Live steam was introduced just above the waste discharge valve and the furfural laden vapour was passed through the vapour outlet valve and an orifice-type flowmeter to a copper condenser. The condensate was collected in a copper tank mounted on platform scales.

(ii) The operating procedure.

The digester was charged with 60 lb. of air-dry oat-hulls and the required quantities of water and sulphuric acid. Steam was introduced into both the digester and the jacket and the required digestion pressure was obtained in about five minutes. To prevent excessive condensation of the live steam in the digester, the jacket pressure was adjusted to exceed the digestion pressure by 25 lb./sq. in. throughout the running and blowing-down periods.

The vapour outlet valve was adjusted to give the required condensate rate and the digestion pressure was maintained constant by controlling the live steam input. On completion of the digestion the digester pressure was reduced to atmospheric over a period of 30 to 60 minutes and the spent material discharged. Samples of the aqueous condensate were taken at intervals during the digestion and subsequently analysed to determine the rate of furfural production; the total product was sampled to determine the over-all yield.

(iii) Analytical methods.

Furfural.—The furfural content of aqueous furfural solutions was determined by a modification of the method of Hughes and Acree (21).

About 150 g. of crushed ice, or sufficient to maintain the temperature of the solution at 0°C. during the titration, is placed in a 500 ml. iodine flask and a 25 ml. aliquot of the furfural solution containing from 50 to 100 mg. of furfural added. To this mixture is added 25·00 ml. of decinormal potassium bromate solution containing 50 g. per litre of potassium bromide, the ice-cold mixture is quickly acidified with 17 ml. of concentrated hydrochloric acid, and the stoppered flask is shaken and allowed to stand for from five to seven minutes with occasional shaking. After the addition of 10 ml. of an aqueous solution of potassium iodide containing 150 g. per litre, the mixture is titrated with decinormal sodium thiosulphate. One ml. of decinormal potassium bromate is equivalent to 0·00480 g. of furfural.

The method was checked using solutions made from furfural carefully purified by the method of Evans and Aylesworth (22) and the results compared with those obtained using the gravimetric thiobarbituric acid method of Mackney and Reynolds (23). The results of the comparison are shown in Table 1.

TABLE 1.—A COMPARISON OF ANALYTICAL METHODS FOR FURFURAL.

Mg. Furfural per Aliquot.	Weight Percentage of Furfural.			Error in Bromate Estimation.
	As Weighed.	Gravimetric Method.	Bromate Method.	
				%
10·36	1·036	1·045	1·092 ± 0·112	± 10
27·00	2·695	2·610	2·736 ± 0·138	± 5·4
51·80	1·036	1·045	1·047 ± 0·011	± 1·0
67·38	2·695	2·610	2·710 ± 0·025	± 0·92

The errors shown for the bromate method represent the errors due to those of the burette readings made in the standardization of the solutions and in the analysis.

These results show that the bromate method has an accuracy of 1 per cent. when using synthetic solutions of furfural containing at least 50 mg. per 25 ml. aliquot.

Solid raw materials were analysed for furfural by distilling samples with 12 per cent. hydrochloric acid (24) and determining the furfural content of the distillate by the bromate method.

(iv) The study of the process variables.

An indication of the optimum operating conditions for the production of furfural from both oat-hulls and maize-cores was obtained from a series of experiments in which the effect of each of the process variables was studied over a wide range.

The results of this process study are shown in Figs. 1 and 2 and indicate that the best yield of furfural from oat-hulls is obtained when the operating conditions are in the following ranges:—

Catalyst concentration, 1·0 to 1·5 lb. of 93 per cent. H_2SO_4/100 lb. dilute acid.

Working pressure, 120 to 140 lb./sq. in. (gauge).

Liquid/solid ratio, 0·5 to 2·0.

Steam throughput, exceeding 65 lb./(hr.) (100 lb. oat-hulls).

The optimum period of digestion was found to be from 3 to 4 hours at working pressure.

The results obtained with maize-cores were substantially the same.

Throughout this work the raw materials were weighed in an air-dry condition as they were received from storage under cover and all yields are expressed on the air-dry basis. The best percentage recoveries of furfural from oat-hulls and maize-cores were 57·2 and respectively, indicating that factors other than these operating conditions play an important part in determining the yield of furfural. These limiting factors are discussed in an accompanying paper.

PROCESS STUDY WITH OAT-HULLS.

Furfural yield in 3 hour runs.
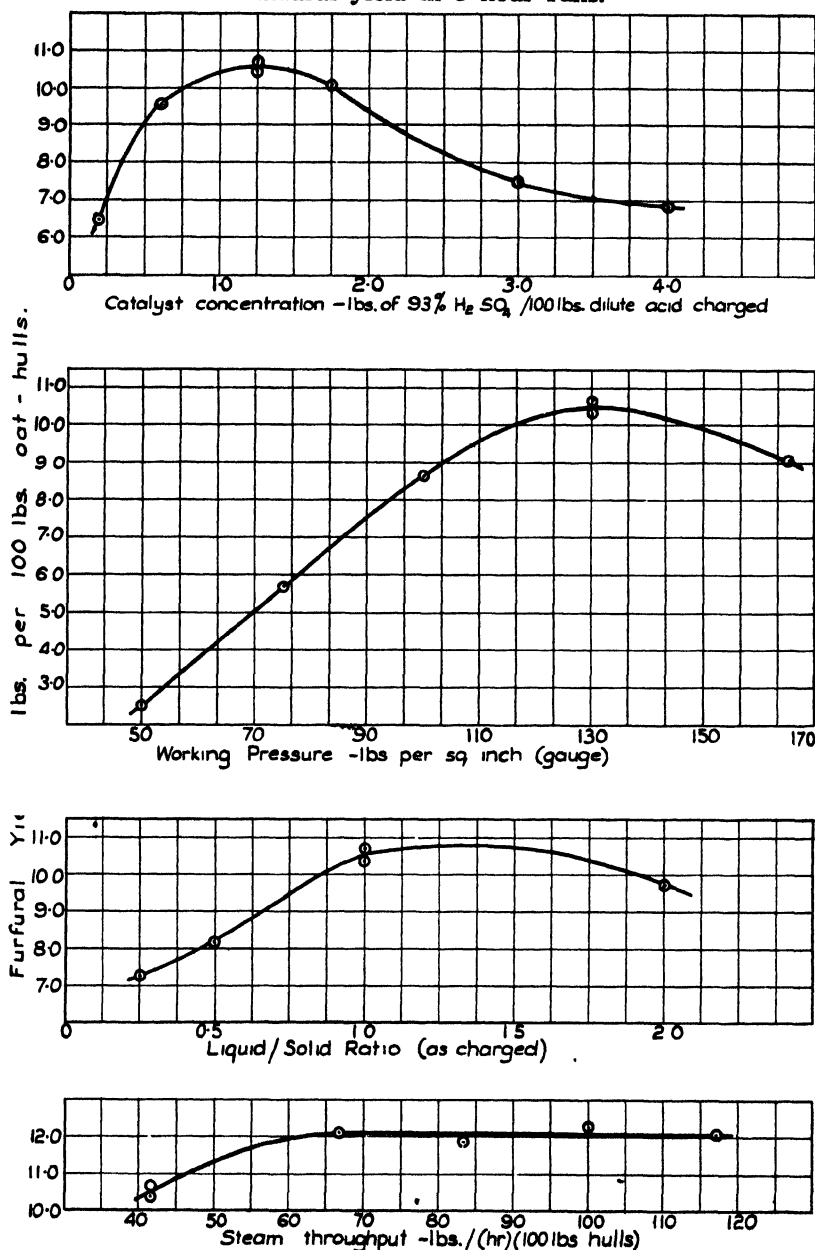
FIG. 1.

Under the optimum conditions for furfural production from oat-hulls 1·4 lb. of acids (expressed as acetic) were produced per 100 lb. of raw material at a concentration of 0·72 per cent. in the condensate,

while the corresponding figures for maize-cores were 2·25 lb./100 at a concentration of 0·6 per cent. The quantity of acetic acid was found to increase with increasing catalyst concentration.

Flax shives were also investigated as a raw material for furfural production but were found to be unsatisfactory because of the relatively poor yield obtained, their low bulk density of 7 lb./cu. ft. and the difficulty encountered in manipulating this long-fibred material. The best yield of furfural obtained from flax shives was from 6 to 7 lb./100 lb. in an aqueous solution containing only from 1·0 to 1·5 per cent. of furfural.

PROCESS STUDY WITH MAIZE-CORES.

Furfural yield in 3 hour runs.
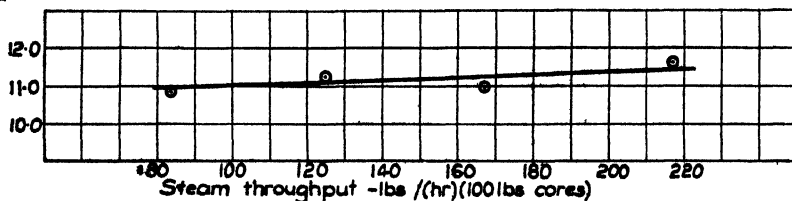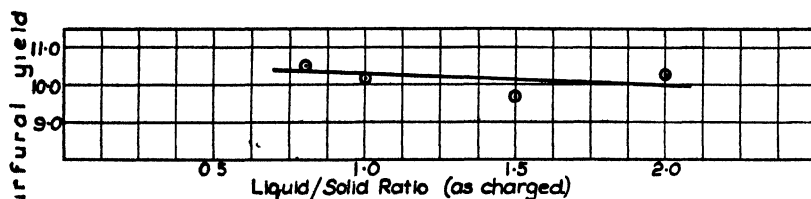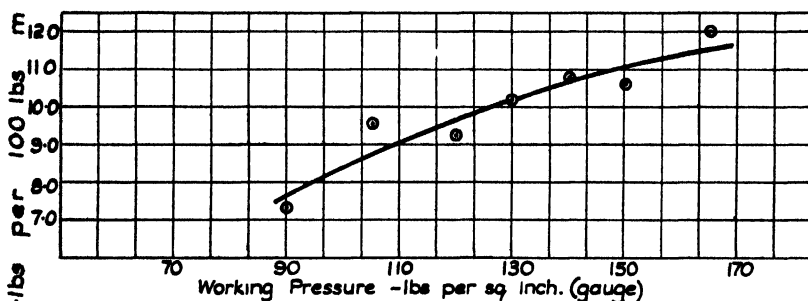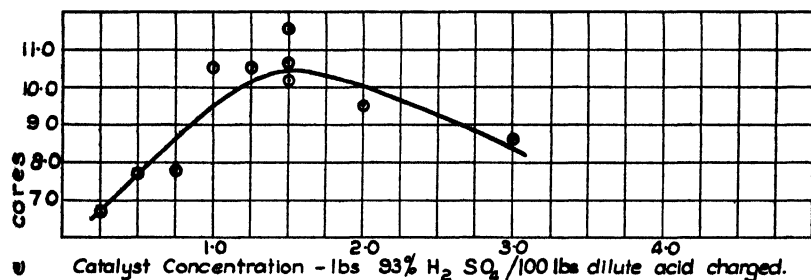
FIG. 2.

(v) The determination of additional data for pilot plant design.

Experience gained during the process study showed that it would be essential to control accurately the flowrate of live steam to the digester and the amount of indirect heat supplied, to maintain a constant liquid/solid ratio throughout the digestion period. It was also found to be essential to provide agitation, to increase the depth : diameter ratio of the digester because of the considerable decrease in the bulk of the raw material in the early stages of the digestion, and to provide a filter for the removal of wax from the condensate.

3. The Pilot Plant Investigation

(i) The pilot plant.

The pilot plant digester and auxiliary equipment, which is illustrated in Plate 1, was designed for the digestion of 300 lb. of oat-hulls per batch and was arranged on three levels. The uppermost level housed the mild steel charging hopper of 30 cubic feet capacity, a 50-gallon copper dilute acid tank, and the condenser which was a horizontal, four-pass, floating-head unit with fifteen $\frac{1}{2}$-in. diameter, 13-gauge copper tubes per pass. The central operating level housed the digester, the instrument panel, the 180 gallon capacity copper condensate tank mounted on platform scales, and a condensate filter. Two copper condensate storage tanks each of 230 gallons capacity were mounted below the operating level and a mobile rectangular mild steel tank of 32 cubic feet capacity was provided at ground level for the removal of the residue discharged from the digester.

The vertical cylindrical digester, 5 feet long and 2 ft. 6 in. in diameter, which was fabricated from $\frac{3}{8}$-in. mild steel plate, was provided with a 3-in. bronze gate valve for discharging, a live steam inlet, and a footstep bearing for the stirrer shaft. The flanged dished cover, which had an 8-in. diameter filling opening, a central stirrer mounting, and a 2-in diameter vapour outlet, was attached to the digester body with forty $\frac{3}{4}$-in. diameter high tensile steel bolts. The body and cover were lined with 10-gauge copper which proved satisfactory throughout this work. Indirect heating was provided by a steam coil of ten turns of 1-in. diameter copper tube spaced at $1\frac{3}{4}$-in. centres and mounted 2 inches from the walls of the digester. The stirrer consisted of three pairs of bronze paddle blades rotating at 32 r.p.m. and driven through a reduction gear by a 3-h.p. electric motor.

All control valves and measuring instruments were situated on the instrument panel. The live steam entering the digester was measured with an orifice flowmeter actuating an electrically indicating mercury manometer. The vapour flow from the digester was controlled from an orifice flowmeter but was measured by direct weighing of the condensate. Pressure, both in the digester and in the steam coil, was determined by calibrated pressure gauges, the volume of condenser-water by a calibrated positive displacement water meter, and the stirrer power consumption by a kilowatt-hour meter.

(ii) Method of operation.

The digester was charged with 300 lb. of raw material and the required quantity of dilute acid. Live steam was admitted at the rate of 300 lb./hour to sweep air from the digester through the filling opening, the digester was then closed and the live steam rate adjusted to permit the attainment of working pressure in from 45 to 60 minutes. Stirring was commenced when the digester pressure reached 40 to 50 lb./sq. in. and, just before the working pressure was reached, the steam pressure in the heating coil was adjusted to exceed the working pressure by 15 lb./sq. in. The live steam inlet and vapour outlet valves were then adjusted to give the desired rate of steam throughput and, during the digestion period, the working pressure was maintained constant by the adjustment of the steam pressure in the heating coil.

At the conclusion of the digestion period the live steam valve was closed and the rate of vapour discharge increased to about 300 lb./hour to reduce the digester pressure to atmospheric in 30 minutes.

Readings of gauges and meters were recorded at five minute intervals and condensate samples were taken when required. The total condensate was weighed and sampled and then allowed to cool overnight before filtering and storing for subsequent distillation.

(iii) Experimental results from the pilot plant investigation.

(a) *The Effect of Variation of Operating Conditions on the Yield of Furfural.*

The experimental results obtained with oat-hulls in runs of three hours duration at working pressure are shown in Table 2 and the results obtained with maize-cores in similar runs are shown in Table 3. These results indicate the variation in yield of furfural which may be expected by varying the conditions of digestion. Results obtained with rice-hulls, hardwood sawdust, and flax shives are compared with those for oat-hulls and maize-cores in Table 4. The operating conditions used in these latter experiments were not necessarily the optimum ones for each material.

(b) *The Effect of the Period of Digestion on the Yield and Concentration of Furfural.*

The digestion period can be divided into three parts, the period required to achieve the working pressure, the period at the working pressure, and the period required to reduce the working pressure to atmospheric or the " blow-down " period.

The results of runs of different duration, using oat-hulls and maize-cores, and having the condensate from the working period and blow-down period collected separately, are shown in Table 5 and in Figs. 3 and 4.

The furfural concentration in the vapour from the digester throughout a typical five-hour run and during the " blow-down " period of a three and four-hour run is shown in Fig. 5.

(c) The Use of Sodium Bisulphate as Catalyst.

It has been suggested (25) that the addition of metallic salts to the digestion mixture might increase the yield of furfural due to their tendency to increase the critical solution temperature of furfural-water mixtures.

Some runs were carried out using sulphuric acid as the catalyst with and without the addition of an equivalent quantity of sodium sulphate. The results of these runs showed that the addition of the

TABLE 2.—RESULTS OBTAINED IN THREE-HOUR RUNS WITH OAT-HULLS.

Run Number.	Catalyst Concentration Percentage H_2SO_4.	Working Pressure lb./sq. in.	Liquid/Solid Ratio as Charged.	Steam Throughput lb./(hr.) (100 lb.).	Furfural.	
					Condensate Concentration Percentage.	Yield lb./100 lb.
B 27	1·0	130	1·5	66·7	3·80	9·9
B 18	1·25	,,	,,	,,	4·00	10·4
B 20	,,	,,	,,	,,	3·90	10·4
B 60	,,	,,	,,	,,	4·15	10·5
B 37	1·5	,,	,,	,,	4·15	10·9
B 66	,,	,,	,,	,,	3·62	9·9
B 85	,,	,,	,,	,,	3·92	9·9
B 87	,,	,,	,,	,,	4·46	11·3
B 39	1·75	,,	,,	,,	3·73	9·3
B 41	1·25	110	,,	,,	3·70	9·5
B 61	,,	,,	,,	,,	4·06	10·1
B 35	,,	120	,,	,,	3·95	9·8
B 36	,,	,,	,,	,,	3·98	9·6
B 55	,,	,,	,,	,,	4·25	10·5
B 56	,,	,,	,,	,,	4·26	11·2
B 57	,,	,,	,,	,,	4·25	10·6
B 18	,,	130	,,	,,	4·00	10·4
B 20	,,	,,	,,	,,	3·90	10·4
B 60	,,	,,	,,	,,	4·15	10·5
B 43	,,	140	,,	,,	3·80	9·6
B 48	,,	,,	,,	,,	3·90	9·9
B 50	,,	,,	,,	,,	3·70	9·6
B 53	,,	,,	,,	,,	4·15	10·8
B 54	,,	,,	,,	,,	3·87	10·1
B 58	,,	,,	,,	,,	4·15	10·8
B 59	,,	,,	,,	,,	4·00	10·5
B 44	,,	150	,,	,,	3·70	9·7
B 46	,,	,,	,,	,,	3·74	10·6
B 51	,,	,,	,,	,,	3·87	10·5
B 52	,,	,,	,,	,,	3·80	10·2
B 34	,,	130	1·25	,,	4·20	10·5
B 18	,,	,,	1·50	,,	4·0	10··
B 20	,,	,,	1·50	,,	3·9	10·4
B 71	,,	,,	1·75	,,	3·7	9·9
B 94	,,	,,	1·75	,,	3·8	9·8
B 30	,,	,,	1·50	58·4	4·28	9·6
B 29	,,	,,	,,	61·6	4·05	10·0
B 18	,,	,,	,,	66·7	4·00	10·4
B 20	,,	,,	,,	,,	3·90	10·4
B 60	,,	,,	,,	,,	3·80	9·9
B 61	,,	,,	,,	,,	4·15	10·9
B 22	,,	,,	,,	75·0	3·65	10·7
B 25	,,	,,	,,	,,	3·75	10·5
B 26	,,	,,	,,	,,	3·60	10·7
B 21	,,	,,	,,	83·4	3·25	10·2

TABLE 3.—RESULTS OBTAINED IN THREE-HOUR RUNS WITH MAIZE-CORES.

Run Number.	Catalyst Concentration Percentage H_2SO_4.	Working Pressure lb./sq. in.	Liquid/ Solid Ratio as Charged.	Steam Throughput lb./(hr.) (100 lb.).	Furfural.	
					Condensate Concentration Percentage.	Yield lb./100 lb.
B 112	0·5	130	1·5	66·7	3·22	7·9
B 111	1·0	,,	,,	,,	3·67	9·4
B 109	1·25	,,	,,	,,	3·48	9·0
B 108	1·50	,,	,,	,,	3·14	8·4
B 129	1·50	,,	,,	,,	3·32	8·4
B 110	1·75	,,	,,	,,	2·64	6·9
B 121	1·50	110	,,	,,	3·41	8·0
B 120	,,	120	,,	,,	3·48	8·1
B 108	,,	130	,,	,,	3·14	8·4
B 129	,,	130	,,	,,	3·32	8·4
B 123	,,	140	,,	,,	3·08	7·3
B 122	,,	150	,,	,,	2·96	7·3
B 113	1·5	130	1·0	,,	2·65	6·2
B 108	,,	,,	1·5	,,	3·14	8·4
B 129	,,	,,	1·5	,,	3·32	8·4
B 114	,,	,,	2·0	,,	2·61	7·2
B 115	,,	,,	1·5	50·0	2·90	5·6
B 116	,,	,,	,,	58·4	3·05	6·6
B 108	,,	,,	,,	66·7	3·14	8·4
B 129	,,	,,	,,	66·7	3·32	8·4
B 119	,,	,,	,,	75·0	3·29	8·4
B 118	,,	,,	,,	83·4	3·03	8·5

TABLE 4.—RESULTS OBTAINED WITH VARIOUS RAW MATERIALS.

Raw Materials and Run Number.	Raw Material.			Operating Conditions.*		Total Furfural Content of Products lb./100 lb.			Yield lb./100 lb.	Percentage Recovery.
	Bulk Density lb./cu. ft.	Contents lb./100 lb.		Catalyst Concentration Percentage H_2SO_4.	Steam Throughput lb./(hr.)×(100 lb.).	Liquid Residue.	Solid Residue.	Condensate.		
		Furfural.	Moisture.							
Oat-hulls B 104 ..	13·2	19·0	9·9	1·5	66·7	0·32	0·78	3·60	9·2	48·5
Maize-cores B 129 ..	11·5	20·3	6·1	1·5	66·7	0·29	0·47	3·32	8·4	41·2
Rice-hulls B 131 ..	8·7	9·8	9·2	1·0	70·0	0·19	0·40	1·74	5·0	51·5
Hardwood sawdust B 133 ..	13·0	7·0	10·8	1·0	50·0	0·10	0·25	1·28	2·9	41·4
Flax shives A 123 ..	7·0	14·0	..	1·5	73·0	..	..	1·44	7·2	51·4

* These runs were 3-hour runs made at a working pressure of 130 lb./sq. in. and with a liquid/solid ratio of 1·5.

Oat-hulls: Variation of furfural yield with digestion time.
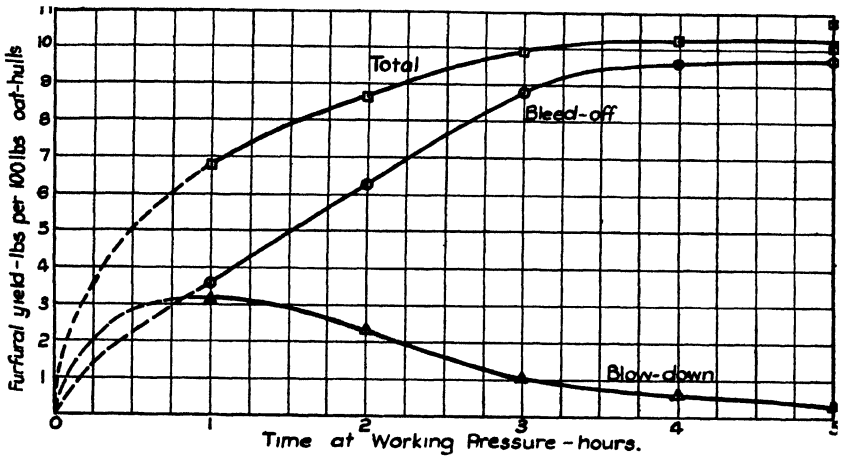
FIG. 3.

Maize-cores: Variation of furfural yield with digestion time.
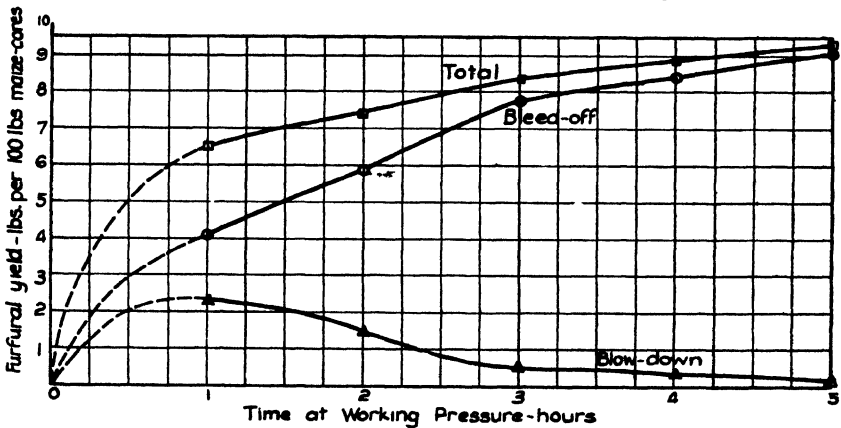
FIG. 4.

Oat-hulls: Variation of furfural concentration with time.
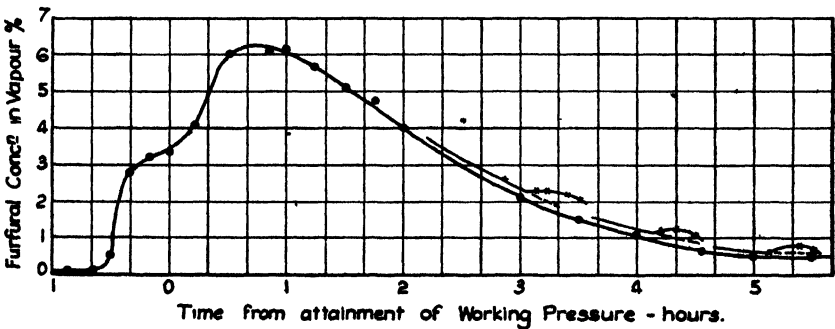
FIG. 5.

TABLE 5.—EFFECT OF DURATION OF DIGESTION ON YIELD OF FURFURAL.‡

Raw Material	Run Number.	Time at Working Pressure.	Product from Period at Working Pressure.			Product from Blow-Down Period.			Yield.	Furfural Concentration.		Lb. Steam Used per Lb. Furfural.*
			Total.	Furfural.		Total.	Furfural.			At End of Blow-down.	In Total Product.	
		hrs.	lb.	%	lb.	lb.	%	lb.	lb./100 lb.	%	%	
Oat-hulls	B 89	1	205	5·27	10·8	155	6·03	9·35	6·7	5·49	5·59	27·3
	B 92	2	392	4·77	18·7	161	4·50	7·25	8·7	4·15	4·68	28·9
	B 66	3	660	4·00	26·4	160	2·05	3·28	9·9	2·00	3·62	32·0
	B 91	4	799	3·60	28·8	159	1·15	1·83	10·2	1·15	3·20	37·6
	B 90	5	1,004	2·90	29·1	147	0·59	0·87	10·0	0·58	2·60	45·0
Maize cores	B 128	1	200	6·12	12·2	169	4·24	7·16	6·5	6·40†	5·25	28·4
	B 134	2	408	4·30	17·6	156	2·98	4·64	7·4	3·50	3·93	33·6
	B 129	3	630	3·70	23·3	128	1·38	1·76	8·4	1·47	3·32	37·9
	B 126	4	797	3·18	25·3	165	0·85	1·40	8·9	0·88	2·78	43·1
	B 124	5	1,016	2·69	27·4	141	0·41	0·58	9·3	0·58	2·42	48·3

* Steam used /lb. furfural calculated on the basis of 200 lb./hr. plus 350 lb. required to heat charge to working pressure.

† For maize-cores these concentrations were at end of working period not at end of blow-down.

‡ The operating conditions used in these runs for oat-hulls and maize-cores were :—Working pressure 130 lb./sq. in., catalyst concentration 1·5 per cent., liquid/solid ratio 1·5, steam throughput 66·7 lb./(hr.) (100 lb. raw materials).

sodium sulphate gave a slight increase (0·5 to 1 lb./100 lb. hulls) in furfural yield over the range of concentration from 1·45 to 4·35 lb. of sodium sulphate per 100 lb. of solution.

Although these tests were not comprehensive they showed that sodium bisulphate could be used as a catalyst, but its advantage over sulphuric acid was not great.

(iv) Discussion of results.

(a) *Catalyst Concentration.*

The best yield of furfural was obtained from oat-hulls, under the conditions specified, with a catalyst concentration of from 1·25 to 1·5 per cent., while a slightly lower concentration of from 1·0 to 1·25 per cent. was the most satisfactory for maize-cores. At a lower catalyst concentration a lower yield was obtained and some difficulty was experienced in discharging the spent mass from the digester, while higher concentrations gave a lower yield, due to increased decomposition of furfural and the intermediate pentoses.

(b) *Working Pressure.*

There was no gain in the yield of furfural from either oat-hulls or maize-cores by increasing the working pressure above 130 lb./sq. in. The use of higher pressures was undesirable as the yield of furfural was decreased owing to an increase in rate of formation of undesirable by-products and to the longer time required to reach working pressure. The use of lower pressures with correspondingly higher catalyst concentrations did not give any improvement in the furfural yield.

(c) *Steam Throughput.*

The yield of furfural from both oat-hulls and maize-cores increased slowly as the steam throughput was increased to about 70 lb./(hr.) (100 lb. raw material) and then remained substantially constant. The use of higher throughputs served only to increase dilution of the aqueous furfural.

(d) *Liquid/Solid Ratio.*

Variation of this factor did not greatly affect the yield of furfural. The best yields of furfural were obtained at a liquid/solid ratio of from 1·25 to 1·5 as charged; values lower than 1·0 produced a thick digestion mixture which was difficult to stir and discharge from the digester, while values higher than 1·75 resulted in increased steam consumption.

(e) *The Period of Digestion.*

The results obtained with oat-hulls indicated that there was no advantage in extending the digestion period beyond three hours and that the period required to reach working pressure should not exceed 30 to 40 minutes. In those cases where the digestion period was extended to five hours the quantity of furfural produced during the fourth and fifth hours was small in relation to the quantity of steam

used. Actually almost equal yields of furfural were obtained for the three-hour digestion because of the greater quantity of furfural then produced during the "blow-down" period.

The results of the runs with maize-cores showed the same trend, but indicated that a slightly longer digestion period was an advantage.

In the shorter runs the low yield was due to incomplete stripping of furfural, while in the longer runs the stripping of the furfural was virtually complete.

(v) The optimum conditions for the production of furfural.

The optimum conditions for the production of furfural from oat-hulls and maize-cores in the plant described in this paper are detailed in Table 6.

There was no indication that further alterations in these variables would increase the yield of furfural.

TABLE 6.—THE OPTIMUM CONDITIONS FOR THE PRODUCTION OF FURFURAL.

—	Oat-hulls.	Maize-cores.
Catalyst Concentration ..	1·25–1·50 per cent. ..	1·0–1·25 per cent.
Working Pressure	120–130 lb./sq. in. ..	120–130 lb./sq. in.
Liquid/Solid Ratio	1·25 to 1·50 ..	1·4 to 1·6
Steam Throughput	65–75 lb./(hr.) (100 lb.)	65–70 lb./(hr.) (100 lb.)
Time of Digestion—		
To Working Pressure ..	30–40 minutes ..	30–40 minutes
At Working Pressure ..	3–4 hours	3–4 hours
Blow-down	30–45 minutes ..	30–45 minutes

(vi) Additional data necessary for the design of digestion plant.

In addition to the determination of the range of operating conditions under which a satisfactory yield of furfural can be obtained, these experiments provided some of the data necessary for the design of larger plant.

Further pilot-plant runs were made to obtain other data necessary for the design of larger digestion equipment.

(a) The Digester Shape.

The optimum shape of digester required for carrying out this process depends on the depth of charge necessary to ensure efficient stripping of the furfural by the live steam and on the relationship between digester diameter and power requirements for stirring.

The effect of charge depth and steam throughput on the rate of stripping of furfural was determined in a series of runs in which the steam throughout and the charge depth was varied and in which samples of the condensate were taken hourly to determine the rate of furfural stripping.

TABLE 7.—THE EFFECT OF CHARGE DEPTH AND STEAM THROUGHPUT ON STRIPPING OF FURFURAL.

Run Number *	Working Depth (feet).	Steam Throughput lb/(hr) (100 lb Hulls)	Furfural Stripping Rate lb/(hr) (100 lb Hulls) at—			Lb Steam Used /lb Furfural Stripped at—			Total Furfural Yield lb/ 100 lb Hulls	Total Steam Used / Total Furfural Yield
			1st Hour	2nd Hour	3rd Hour	1st Hour	2nd Hour	3rd Hour		
B 78	2·4	108	4·64	2·71	1·18	23·3	39·8	91·4	11·0	29·5
B 141	2·6	90·0	4·50	2·76	1·31	20·0	32·3	68·7	9·85	27·4
B 142	2·6	90·0	4·63	2·70	1·27	19·5	33·4	70·9	9·81	27·5
B 143	3·2	90·0	4·74	2·62	.	19·0	34·4	..	9·45	28·6
B 144	3·2	90·0	4·79	2·65	.	18·8	34·0	..	9·90	27·3
B 76	3·4	72·0	4·09	2·88	1·69	17·6	25·0	42·6	9·84	21·9
B 75	3·8	74·7	4·20	2·92	1·64	17·8	25·6	45·6	9·83	22·8
B 82	4·8	66·0	3·76	2·74	1·58	17·6	24·1	41·8	9·36	21·0

* These runs were made with a working pressure of 130 lb/sq in, a liquid/solid ratio of 1·5 and a catalyst concentration of 1·5 per cent

Since the conversion of pentosan to furfural was complete after the first hour* of the digestion period, subsequent changes in the furfural vapour concentration were dependent only on the rate of stripping.

Results of these tests, shown in Table 7, indicate that a substantially constant value is obtained for the ratio of steam used to furfural stripped when the charge depth exceeds 3·5 feet. Therefore a digester of similar shape to that used in the pilot plant would be suitable for larger scale production although a slightly smaller diameter/height ratio would be preferable from the construction and stirrer power requirement aspects.

(b) *Stirring Requirements.*

The effect of stirring on the yield of furfural from oat-hulls was determined from a series of tests, the results of which are shown in *Table 8.*

TABLE 8.—THE EFFECT OF STIRRING ON FURFURAL YIELD.

Run Number.	Stirrer.	Steam Throughput lb./(hr.)(100 lb.).	Furfural.	
			Condensate Concentration Percentage.	Yield lb./100 lb. Oat-hulls.
B 95	Off .. .	66·7	3·12	7·8
B 97	„	83·4	2·93	7·8
B 98	„	88·0	2·64	8·4
B 18	On .. .	66·7	4·00	10·4
B 20	„	66·7	3·90	10·4

These results showed that stirring was necessary and that the agitating effect of the live steam was small, even at relatively high throughputs. The principal function of the stirrer was to overcome the channelling of the steam. The power consumption of the stirrer at various stages during the digestion is shown in Table 9.

These results showed that the stirrer power requirements were substantially the same for all the raw materials used and that there was a rapid drop in the power consumption during the early stages of the digestion. As no significant loss in yield was obtained when the stirrer was started just before the working pressure was reached, stirring in the early stages would be unnecessary in a production plant.

(c) *Digester Condenser Requirements.*

(1) *Pilot plant condenser.*—Preliminary runs with the pilot plant indicated that the over-all heat transfer coefficient for the condensation of the aqueous digestion product was much smaller than that for steam because a substantial quantity of wax from the product was deposited on the condenser tubes, giving a low vapour-side film coefficient which was, in this case, the controlling factor in the heat transfer.

* This *Journal*, p. 258.

Material and heat balances were made over the pilot plant condenser after the plant had been in operation for some time and the wax deposit had reached a maximum to enable the combined vapour-side film and fouling coefficient to be calculated for use in the design of condensers for a larger furfural plant.

TABLE 9.—THE STIRRER POWER REQUIREMENTS.

Raw Material	Oat-hulls.			Maize-cores.	Hardwood Sawdust.	Rice-hulls.
Run Number	B 102	B 103	B 130	B 126	B 133	B 131
Digester pressure at which stirrer started (lb./sq. in.) ..	Start	60	130	110	70	80
	watts.	watts.	watts.	watts.	watts.	watts.
Stage of Digestion—						
Dry oat-hulls ..	2,160	..	..	..	..	..
Wet oat-hulls ..	2,268	..	..	..	..	..
Pressure—						
1 lb./sq. in. ..	1,435	..	..	.	..	..
60 lb./sq. in. ..	600	1,812	..	..	..	..
130 lb./sq. in. ..	370	550	468	468	504	486
After 1 hr. at W.P.	330	360	432	320	396	360
„ 2 hrs. at W.P.	310	340	360	320	396	360
„ 3 hrs. at W.P.	310	330	300	320	396	324
Total power consumption K.W.H. per run	2·24	1·60	1·20	1·17	1·35	1·28

Accurate calculated values of the vapour-side film coefficient for condensation could not be obtained from the experimental data on the pilot plant condenser, as this four water-pass unit acted as a de-superheater and cooler as well as a condenser. Approximate values could be calculated, however, by assuming single pass operation and first determining the relative tube areas required for de-superheating, condensing, and cooling.

Under favourable operating conditions, with a water exit temperature of about 200°F., the vapour-side film coefficient for condensation of dilute furfural vapour from the digester was found to be of the order of 200 ± 50 B.T.U./(hr.) (sq. ft.) (deg. F.).

(2) *The effect of partial wax removal.*—Runs were also made with an experimental, horizontal, single-tube condenser to determine the effect of partial wax removal by de-superheating the vapour before condensing it. The experimental unit was operated with vapour from a normal digestion, starting with a clean tube and proceeding until constant fouling conditions were obtained as shown by the vapour-side film coefficient remaining substantially constant for subsequent runs.

There was an initial rise in the value of the vapour-side film coefficient from 1220 ± 50 B.T.U./(hr.) (sq. ft.) (deg. F.) to 1430 ± 65 which was probably due to the promotion of dropwise condensation by a thin wax film on the tube surface. This was followed

by a rapid fouling of the tube and a simultaneous fall in the value of the film coefficient to 800 ± 30 B.T.U./(hr.) (sq. ft.) (deg. F.) at which value it remained substantially constant.

(d) The Effect of Indirect Heating.

A steam coil was used to provide indirect heating in the pilot plant to compensate for heat losses and to maintain the liquid level constant during the digestion. The use of indirect steam was found to have no appreciable effect on the yield of furfural and would be unnecessary in a larger production unit.

(e) Material Balance Runs.

Material balances were made when operating with oat-hulls under the following operating conditions: working pressure 130 lb./sq. in., catalyst concentration 1·5 per cent., liquid/solid ratio 1·5 and a steam throughput 66·7 lb./(hr.) (100 lb. hulls), with a blow-up time of 52-60 minutes, digestion time of three hours, a blow-down time of 30-35 minutes, and a high water velocity through the condenser to prevent furfural loss from the condensate. The condenser water consumption was calculated on the basis of a water temperature rise of 140 deg. F. and a condensate exit temperature of 210° F. Results are shown in Table 10. The " lignin " values were determined as percentage insoluble in 72 per cent. sulphuric acid (26).

These material balance figures showed clearly what takes place during the digestion process and indicated the quantities of raw materials and utilities required for furfural production. The increase in the " lignin " content of the solid material was due to tarry decomposition products. The wax in the product was found to contain 95 per cent. of fatty acids consisting chiefly of palmitic and oleic acids.

4. The Recovery of Furfural from the Digestion Product

Furfural is most readily recovered from the aqueous solutions obtained from the digestion process by means of distillation, although the solvent extraction methods which have been described (27, 28, 29) may prove more economical for more dilute solutions.

Distillation methods are attractive economically because of the enrichment which occurs when the azeotrope, containing 35 per cent. furfural, separates on cooling into two layers, the lower of which contains 95 per cent. furfural at atmospheric temperature. Pure furfural can be obtained by subsequent distillation of the 95 per cent. product under reduced pressure.

(i) The distillation of aqueous furfural solutions.

The distillation of aqueous furfural solutions has been described by La Forge and Mains (9) and equipment for this separation employing both vapour and liquid feeds has been described (14, 15, 30).

The aqueous furfural solutions obtained from the digestion process normally contain from 2 to 5 per cent. furfural, 0·5 to 1·5 per cent. acids (chiefly acetic) and about 0·05 per cent. of volatiles (chiefly methanol). When the mixture is continuously distilled in a suitable column the furfural water azeotrope, containing 35·0 per cent. furfural and boiling at 208°F., is obtained as a two-phase distillate

TABLE 10.—RESULTS OF MATERIAL BALANCE DIGESTIONS.

Run Number.	Charge.							Steam.				Stirrer Power Consumption.*	Condensate.					
	Water.	Oat-hulls.	93% H₂SO₄.	"Lignin" in Hulls.	"Lignin."	Potential Furfural in Hulls.	Potential Furfural.	Blow-up.	Bun.	Coll.	Total.		Weight.	Furfural.	Acetic Acid.	Furfural.	Acetic Acid.	Water.
	lb.	lb.	lb.	%	lb.	%	lb.	lb.	lb.	lb.	lb.	kw. hrs.	lb.	%	%	lb.	lb.	lb.
B 102	450	300	6·75	..	..	19·1	57·2	350	603	195	1,148	2·24	761	3·62	0·68	27·6	5·2	728
B 103	450	300	6·75	9·73	29·2	19·0	56·9	350	602	210	1,162	1·67	754	3·65	0·70	27·5	5·3	721
B 104	450	300	6·75	9·73	29·2	19·3	58·0	350	603	201	1,154	1·86	765	3·60	0·65	27·6	5·0	732

* In Run B 102 the stirrer was on from start, in other runs from when digester pressure was 60 lb./sq. in.

Run Number.	Wax in Product.	Liquid Residue.				Solid Residue.						Requirements for Digestion per lb. Furfural.					
		Total Residue.	Wt.	Potential Furfural.	Furfural.	Wet Wt.	Dry Wt.	"Lignin."	Potential Furfural.	"Lignin."	Potential Furfural.	Water.	Steam.	Water.	Power.	Oat-hulls.	93% H₂SO₄.
	lb.	lb.	lb.	%	lb.	lb.	lb.	%	%	lb.	lb.	lb.	lb.	lb.*	kw. hr.	lb.	lb.
B 102	1·25	902	572	0·45	2·5	330	175	..	0·38	..	0·67	155	41·6	217	0·081	10·9	0·25
B 103	..	913	522	0·34	1·9	391	171	49·3	0·78	84·4	1·3	220	42·3	217	0·061	10·9	0·25
B 104	..	894	595	0·30	1·8	299	154	50·4	0·79	77·5	1·2	145	41·8	217	0·063	10·9	0·25

* 200 lb. condenser water and 17 lb. charge water.

which when cooled to 72°F. gives layers consisting of solutions containing 95 and 8·4 per cent. furfural. The aqueous layers can be returned to a suitable point in the column above the feed inlet, but if this is done, it is necessary to remove the volatiles which would otherwise accumulate in the upper part of the column and in the decanter and thereby increase the mutual solubility of the two layers. Almost all the acetic acid is contained in the aqueous waste from the column.

(a) Distillation Column Design Methods.

(1) *Mains method.*—The only method described for the design of distillation equipment specifically for the recovery of furfural from its aqueous solutions is that of Mains (12) who extended the method of Lewis (31) to take into account the additional liquid feed returned to the column from a continuous decanter.

(2) *The application of McCabe-Thiele method to the distillation of furfural solutions.*—The graphical McCabe-Thiele (32) method was applied to the design of furfural distillation equipment to determine its usefulness for this system, as the Mains method is tedious. A comparison of results obtained by the two methods is shown in Table 11. The calculations in the McCabe-Thiele method were made using the vapour-liquid equilibrium data of Mains (10) and the assumptions that the waste composition was 2 per cent. of that of the feed, that closed steam heating was employed, that the feed, reflux, and decant entered the column as liquids at their boiling points, and that the operation was under conditions of constant molal overflow. The last-mentioned assumption was justified as the molal latent heats of vaporization of furfural and water at their boiling points are 18,580 and 17,460 B.T.U./lb. mole respectively.

TABLE 11.—A COMPARISON OF McCABE–THIELE AND MAINS
METHODS OF COLUMN DESIGN.

Feed Wt. Percentage Furfural.	Reflux Ratio at Top of Column.	Number of Theoretical Plates Required.					
		Lower Section.		Middle Section.*		Upper Section.	
		McCabe.	Mains.	McCabe.	Mains.	McCabe.	Mains.
1	8	>12	..	1·7	..	1·7	..
	7	..	8·5	..	0·6	..	1·8
2	5	5·7	..	1·2	..	1·7	..
	4	7·1	5·7	1·2	0·5	1·7	1·8
	3	11	11	1·6	0·5	1·7	1·8
	2·5	high	19	2·1	0·7	1·7	1·8
3	4	4·5	2·8	0·9	0·3	1·7	1·8
	3	5·3	3·9	1·0	0·3	1·7	1·8
	2	10	..	1·3	..	1·7	..
	1·5	high	16	2·2	1·0	1·8	1·8
4	4	3·9	1·9	0·4	0·2	1·7	1·8
	3	4·4	2·3	0·6	0·2	1·7	1·8
	2	5·7	..	0·8	..	1·8	..
	1·5	7·6	5·8	1·0	0·4	1·8	1·8

* Between feed inlet and decant return.

When live steam heating is employed, additional plates are required in the stripping section of the column. This additional requirement was calculated by the McCabe-Thiele method, assuming one mole of steam used per mole of furfural solution vaporized; and was found to be from 0·2 to 0·4 theoretical plates depending on the ratio of the liquid to vapour flowrates in the stripping section.

(3) *Experimental distillation of furfural solutions.*—Furfural was recovered from the digestion product by distillation either in a 6-in. diameter column packed with ½-in. Raschig rings or in an 8-in. diameter bubble-cap column.

Short trial runs at various reflux ratios with the packed column using a 3·5 per cent. furfural feed showed the H.E.T.P. to be approximately 24 inches based on the number of theoretical plates determined by the McCabe-Thiele method. This high value was due to the high vapour velocities which were about 85 per cent. of the flooding velocity.

The results of four preliminary experiments with the bubble-cap unit operated as a stripping column by the method of Koffolt (33) with a 1·0 per cent. furfural solution as feed and with a waste containing 0·02 per cent. furfural enabled the approximate number of theoretical plates required at various liquid/vapour ratios to be calculated; as shown in Table 12.

TABLE 12.—EXPERIMENTAL RESULTS WITH BUBBLE-CAP COLUMN.

Run Number.	Liquid/Vapour Ratio.	Number of Theoretical Plates.		
		Experimental.	Calculated.	
			McCabe-Thiele.	Mains.
S-7	4·76	10	6·8	4·0
S-4	4·67	9	6·4	3·7
S-5	4·27	6	5·7	3·0
S-6	3·91	5	5·0	2·5

These results indicate that the McCabe-Thiele method is more satisfactory for the design of furfural distillation equipment than the method used by Mains. It is intended to continue work on certain aspects of the distillation of furfural-water mixtures.

(b) *A Comparison of Liquid and Vapour Feed to the Column.*

The use of the digestion product as a direct vapour feed to the column has been employed (14, 15) as an alternative to the condensed product as a liquid feed. A comparison of these two methods was made to determine their relative advantages.

(1) *The comparison for saturated vapour.*—The minimum reflux ratio and the number of theoretical plates at various higher reflux ratios were calculated by the McCabe-Thiele method for both liquid feeds at the boiling point and saturated vapour feeds containing from

3 to 8 per cent. furfural, using live steam and a waste furfural concentration of 1·6 per cent. of that in the feed. From these results suitable reflux ratios were chosen for each feed concentration such that the total number of theoretical plates required was approximately eight. The essential difference between the two methods is that the minimum reflux for vapour feed is from 9·3 to 13·6 times that for liquid feed, owing to the large and relatively sudden change in slope of the equilibrium curve and to the low mole fraction of furfural in the feed.

Material balances were then calculated on the basis of 10 lb. mol./hour passing through the decanter. For these calculations it was assumed that the vapour from the digester was stripped of its wax before passing to the digester condenser or vapour feed column and also that the live steam used in the digester was equal in quantity to the water in the feed to the columns. The condenser water requirements were calculated on a basis of a water temperature rise of 130 deg. F., and the condenser surface areas were determined on a basis of a water velocity of 5 ft./sec. and vapour-side film coefficients of 800 and 1,300 B.T.U./(hr.) (sq. ft.) (deg. F.) for the digester and column condensers respectively.

The column diameters were determined by the method of Bain and Hougen (34) assuming water as the working substance, 1-in. rings as packing and an operating vapour velocity of 70 per cent. of the flooding velocity. The packed volumes were calculated using an H.E.T.P. of 24 inches.

The results of this comparison are shown in Table 13.

(2) *The effect of superheated vapour on the comparison.*—If the vapour from the digester is superheated to the extent of 70 deg. F., the water requirements for the digester condenser in the case of liquid feed would be increased by 3·6 per cent. and the surface area by 17 per cent. This degree of superheating, in the case of a vapour feed, would change the required reflux ratio for a given number of plates by only 5 per cent., would reduce the column steam requirements by 3·5 per cent., and the effect on the required column diameter would be small.

In a liquid-fed column, equipment for the removal of wax from the digester vapour before its condensation is unnecessary provided adequate condenser capacity is installed. Thus, the cost of a wax stripper for the vapour-fed column is counterbalanced by the cost of additional condenser surface area for the liquid-fed column. The additional digester condenser capacity required can be seen in Table 14.

(3) *Conclusions.*—These calculations show that the use of vapour feed slightly decreases the steam and condenser water consumption, decreases the necessary condenser surface area, but increases the diameter of the distillation column and requires the careful removal of waxy materials with an additional scrubbing column. On a basis of cost there would be little difference between the two methods; the liquid-fed unit would be preferable because of its greater flexibility and ease of operation.

TABLE 13.—COMPARISON OF REQUIREMENTS FOR LIQUID AND VAPOUR FEED TO COLUMN.

Concentration of Furfural in Feed. Wt. %	Minimum Reflux Ratio.		Working Reflux Ratio.		Theoretical Plates req. Upper Sections.		Lower Section.		Steam Required—lb./hr. Digester.	Column.		Total.		Condenser Surface Area sq. ft. Digester.	Column.	Total.	
	L.	V.	L.	V.	L.	V.	L.	V.	L. and V.	L.	V.	L.	V.	L.	L.	L.	V.
3	1·50	14·2	3·0	15·0	2·7	3·6	5·7	5·7	2,400	720	460	3,120	2,860	95·5	22·4	118	86
4	0·95	10·2	2·0	12·0	2·5	3·4	6·1	4·2	1,800	540	530	2,340	2,330	71·8	16·1	88	70
5	0·80	7·9	1·5	9·0	2·4	3·4	5·6	4·7	1,420	450	360	1,870	1,780	57·2	13·4	71	54
6	0·50	6·5	1·5	7·0	2·3	3·5	5·4	5·5	1,160	450	260	1,610	1,420	47·8	13·4	61	43
7	0·40	5·4	1·0	6·0	2·0	3·2	4·7	4·4	990	360	250	1,350	1,240	41·0	10·8	52	38
8	0·33	4·5	1·0	5·0	2·0	3·2	5·0	4·2	840	360	210	1,200	1,050	34·4	10·8	45	32

Concentration of Furfural in Feed. Wt. %	Condenser Water—gal./hr. Digester.	Column.	Total.		Column Diameters—in. Upper Sections.		Lower Section.		Packed Volume—cu. ft. of 1-in. Rings. Upper Sections.		Lower Section.		Total.	
	L.	L.	L.	V.	L.	V.	L.	V.	L.	V.	L.	V.	L.	V.
3	1,850	544	2,390	2,160	13·5	27·0	13·8	11·0	5·4	28·8	11·9	7·5	17·3	36·3
4	1,390	405	1,795	1,760	11·7	24·4	12·0	11·9	3·7	22·0	9·5	6·5	13·2	28·5
5	1,110	338	1,450	1,350	10·7	21·3	10·8	9·8	3·0	17·0	7·3	5·0	10·3	22·0
6	925	338	1,260	1,090	10·7	19·2	10·8	8·3	2·9	14·3	6·7	4·0	9·6	18·3
7	795	270	1,070	950	9·6	17·9	9·8	8·2	2·0	11·0	4·9	3·2	6·9	14·2
8	670	270	940	810	9·6	16·6	9·8	7·5	2·0	9·5	5·2	2·6	7·2	12·1

**TABLE 14.—THE EFFECT OF WAX REMOVAL ON CONDENSER
REQUIREMENTS FOR LIQUID FEED.**

Furfural Concentration in Feed.	Surface Area of Digester Condenser (sq. ft.).	
	Wax Removed (h_g = 800).	Wax Not Removed (h_g = 200).
3 per cent.	95·5	266
4 ,,	71·8	200
5 ,,	57·2	159
6 ,,	47·8	133
7 ,,	41·0	114
8 ,,	34·4	96

h_g = vapour-side film coefficient (including fouling).

(c) Hot Versus Cold Decantation.

A considerable reduction in cooler surface area and cooling water can be realized by decanting in two stages. The 35 per cent. distillate is first decanted at 210°F. to yield a decant containing 18·4 per cent. furfural which is returned to the column and a 84 per cent. product which is cooled to atmospheric temperature and again decanted. The quantity of 8 per cent. decant from this second operation is small and it can be returned cold to the column without effecting its operation.

(d) Removal of the Volatiles.

The nature of the more volatile by-products from oat-hulls was deduced from the boiling points and refractive indices of fractions obtained on distillation of samples from the column decanter and from the vent of the digester condenser. These deductions were confirmed by measuring the melting points of prepared derivatives. The relative proportion of the substances identified was estimated from the distillation curve to be as follows:—

Substance Identified.	Approximate Proportion by Volume.
	%
Methanol ..	80
Acetone ..	15
Acetaldehyde	2
Furane ..	2
Methyl furanes	1

The actual quantity of volatiles present in the digestion product is of the order of 0·05 per cent. The volatiles must be removed at some stage in the process to prevent their accumulation in the upper part of the column and in the decanter. Three methods for their removal have been described: the separation of the volatiles from the vapour by partial condensation(9), separation by partial condensation with decantation (14, 15), and stripping of the volatiles from the aqueous decanter layer in a small auxiliary column before

TABLE 16.—HEAT TRANSFER COEFFICIENTS FOR CONDENSATION OF
FURFURAL-WATER AZEOTROPE.

Run Number	—	D-7.	D-8.	D-10.	D-11.
Water temperature rise	Deg. F. ..	45·1	50·1	45·7	54·3
Water velocity ..	Ft./sec. ..	2·08	2·08	2·08	2·08
Logarithmic-mean temp. difference	Deg. F. ..	120·7	121·2	121·9	116·6
Over-all coefficient U	B.T.U./(hr.) (sq. ft.) (deg. F.)	340	375	343	423
Water-side film co-efficient h_w	B.T.U./(hr.) (sq. ft.) (deg. F.)	622	617	620	632
Absolute percentage error in h_s	. .	13·6	20·0	13·8	38·8
Probable percentage error in h_s	.. .	5·45	8·04	5·61	15·6
Type of condensation	.. .	filmwise	semi-dropwise	filmwise	semi-dropwise
Percentage furfural in condensate	Main .	34·0	35·8	28·3	36·0
	Excess ..	34·9	34·7	30·5	35·0
Vapour-side film co-efficient h_s	B.T.U./(hr.) (sq. ft.) (deg. F.)	1,300 ±70	2,080 ± 170	1,340±70	4,350 ±680

5. Cost of Furfural Production

(i) The demand for furfural in Australia.

The annual consumption of furfural during the last few years has been estimated to be from 10 to 30 tons, used chiefly for modifying phenolic resins. The probable annual demand for plastics manufacture in the near future has been estimated to be from 50 to 100 tons; a maximum of 50 tons as a modifier for phenolic resins and from 10 to 50 tons for the manufacture of phenol-furfural resins. For every 100 lb. of the furfural resin made, 55 lb. each of phenol and furfural would be required as compared with 90 lb. of phenol and 7½ gallons of 40 per cent. formaldehyde. For the raw material costs of these two types of resin to be the same, the wholesale cost of furfural would be 1s. 9d. per lb. calculated on the present costs of phenol at 1s. 6d. per lb. and formalin at 6s. per gallon. If a local supply of furfural was available, additional quantities would be used for the manufacture of derivatives and for use as solvents.

TABLE 17.—LOCATION OF RAW MATERIALS IN AUSTRALIA.

Raw Materials	State	Source of Information	Tons Raw Material Per Year	Equivalent as Furfural Tons Per Year	Location of Milling Plants
Oat-hulls	Victoria	Government Statist	3,500	350	All in Melbourne—several plants
	New South Wales	Various manufacturers	4,500	450	All in Sydney—several plants
Maize cores	Atherton Tableland, Queensland	Queensland Department of Agriculture	2,700	270	At one centre
	Rest of Queensland	Queensland Department of Agriculture	15,400	1,540	On individual farms
	New South Wales	Year Book	9,700	970	Probably on farms
	Victoria	Maize Marketing Board	800	80	On individual farms
Cottonseed hulls	Queensland	Queensland Department of Agriculture	1,000	95	At one mill
Rice hulls	New South Wales	Government Statist	3,750	225	90 per cent. milled in Sydney
	Victoria	Various manufacturers	1,700	102	All in Melbourne
Flax-shives	Victoria		3,500	245	At various centres
	South Australia		2,500	175	At various centres

In addition to these raw materials, a large quantity of bagasse is available in Queensland and New South Wales with a furfural yield of about 8 5 lb./100 and also vast quantities of wood waste are available with a furfural yield of from 2 to 3 lb /100.

(ii) Availability of raw materials.

The quantities of raw materials available in various states based on average values for the years 1940 to 1945, are shown in Table 17. States with production of less than 1,000 tons of individual raw materials are not listed and the figures are only approximate as considerable seasonal variation occurs.

This survey showed that sufficient high grade raw material was available at each of a number of individual centres to supply Australian furfural requirements for some years.

No attempt has been made to determine cost of raw materials other than that of oat-hulls, which at the time of writing was £1 14s. per ton at mills in Melbourne. The supply and cost of the higher grade raw materials would vary with the season and also with the demand for them as stock feed, especially in dry seasons. The cost of other raw materials is probably appreciably less than that of oat-hulls.

(iii) Cost of production.

(a) Design of Plant for Costing Furfural Production.

The present minimum economic scale for furfural production in Australia by methods similar to that investigated is probably of the order of from 100 to 200 tons per year. For the purpose of determining the cost of production from oat-hulls, a plant was designed for a production of 200 short tons per year (400,000 lb.). Details of this plant are given in Table 18, in which the numbers refer to items on the flow sheet.* The plant design was based on the operation of three eight-hour shifts per day for 250 days per year assuming a yield of 10 lb. per 100 lb. of air-dry hulls and an average furfural concentration of 4 per cent. in the digestion product.

(1) *The digestion equipment.*—A digester similar to that used in the pilot plant work to be employed and operated with a working pressure of 130 lb./sq. in., a catalyst concentration of $1 \cdot 25$ per cent., a liquid/solid ratio of $1 \cdot 5$, and a steam throughput of $66 \cdot 7$ lb./(hr.) (100 lb. hulls). The five-hour operating cycle to be divided as follows: charging, 15 mins.; blow-up to working pressure, 30 mins.; at working pressure, 180 mins.; blow-down, 60 min.; and discharge, 15 mins.

(2) *The distillation equipment.*—The 4 per cent. furfural solution from the digester to be concentrated in a continuous, packed column with hot liquid feed and employing a hot decanter and operating with live steam, at a reflux ratio of $1 \cdot 1$ (equivalent to $2 \cdot 0$ with a cold decanter) and with waste furfural concentration of $0 \cdot 03$ per cent.

The 95 per cent. product from this column to be dried to $99 \cdot 9$ per cent. in a continuous packed column operating with closed steam at a 28-in. vacuum (50 mm./Hg.) and at a reflux ratio of $5 \cdot 0$.

The volatiles to be removed in a small packed column from the aqueous stream from the hot decanter before its return to the main column.

The distillation columns were designed by the McCabe-Thiele method (32) using 30 inches for the H.E.T.P. of 1-in. Raschig rings

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TABLE 18.—DETAILS OF PLANT DESIGNED FOR COSTING FURFURAL PRODUCTION.

Number.*	Item.	Size.	Type.	Dimensions.	Materials.	Requirements.		
						Steam lb./day.	Cooling Water gal./day.	Power kw.hr./day.
1	Hull storage	160,000 cu. ft.	Silo		Concrete	..	..	..
2	Hull elevator	250 cu. ft./hr.	Pneumatic		Mild steel	..	..	8
3	Hull hopper	250 cu. ft.			"	..	..	..
4	Strong acid tank	50 gal.	Cylindrical	2'φ x 2'6"	Copper	..	..	..
5	Dilute acid charge tank	500 gal.	"	4'φ x 6'6"	Mild steel copper lined	..	..	80
6	Digester	250 cu. ft.	Vertical	5'φ x 16'	Mild steel copper lined	40,450	..	..
7	Residue storage tank	400 cu. ft.	Cylindrical	9'φ x 5'	Mild steel	..	..	..
8	Residue transport tank	150 cu. ft.	Rectangular	6'x 5'x 5'	"	..	..	..
9	Digester condenser	2,200 lb. vapour/hr.	4-pass, floating head	300 sq. ft.	Copper	..	31,280	..
10	Aqueous furfural storage tanks	2,000 gal. each (2)	Cylindrical	6'φ x 11'6"	Mild steel copper lined	..	..	..
11	Wax filters	50 gal. each (2)	Sand and pebbles		Copper	..	..	..
12	Column feed pumps	2,000 lb./hr. 60 ft. head each (2)	Centrifugal	2'φ x 2'6"	Bronze	..	..	24
13	Feed heater	2,000 lb./hr. 130 deg. F.	4-pass, floating head	15 sq. ft.	Copper	5,430	..	..
14	Concentration column	1,700 lb./hr. feed	Packed	1'φ x 28'	Mild steel copper lined	11,450	..	..
15	Column condenser	666 lb. vapour/hr.	4-pass, floating head	22 sq. ft.	Copper	..	8,620	..
16	Reflux divider		Weir		Bronze	..	..	..
17	Hot decanter	60 gal.	Automatic	2'φ x 3'	Copper	..	250	..
18	Volatiles column	237 lb./hr. feed	Packed	3'φ x 4'	"	240	..	..
19	Strong furfural cooler	90 lb./hr., 135 deg. F.	Concentric tube	3 lengths, 3' of ¾" in ⅞"	"	..	1,310	..
20	Cold decanter	10 gal.	Automatic	1'φ x 2'	"	..	..	..
21	Drying column	70 lb./hr. feed	Packed	5"φ x 6'	"	600	..	..
22	Drying column condenser	40 lb./hr. vapour	2 pass	3.2 sq. ft.	"	..	2,700	..
23	Distillate receivers	20 gal. each (2)	Cylindrical	1'6"φ x 2'	"	..	..	..
24	Return pump	50 lb./hr., 60 ft. head	Gear		Bronze	..	..	1
25	Product cooler	70 lb./hr., 110 deg. F.	Concentric tube	3 lengths of ¼" in ⅜"	Copper	..	166	..
26	Product filter	5 sq. ft.	Cylindrical	1'φ x 2'	Mild steel	..	..	..
27	Product receivers	80 gal. each (2)	"	2'φ x 4	Bronze	..	..	..
28	Vacuum pump	28" Vac. 50 c.f.m.	Hytor (2-stage)			..	..	48
29	Methanol receiver	10 gal.	Cylindrical	1'φ x 2'	Copper	..	..	..

* The numbers refer to items illustrated in folder at end of Journal.

and 12 inches for that of ⅜-in. rings, while the column diameters were determined by the method of Bain and Hougen (34) assuming operation at 70 per cent. of the flooding velocity.

(3) *Condensers and coolers.*—The size and construction of these units and their water requirements were determined on the basis of water temperature rises of from 70° to 200°F. for the digester and big column condensers, from 70° to 90°F. for the drying column, and from 70° to 80°F. for the coolers. Liquid-side coefficients were calculated by the expression

$$h_w = 150 \ (1 + 0 \cdot 011t) \ v^{0 \cdot 8}/D^{0 \cdot 2}$$

where t is the average liquid temperature, v its velocity in ft./sec. and D the inside tube diameter in inches. The vapour-side film coefficients used were those determined from the pilot plant study, namely 200 B.T.U./(hr.) (sq. ft.) (deg. F.) for condensation and 50 for de-superheating in the digester condenser, 1,300 for the column condensers, and 3,000 for steam in the feed heater.

(4) *Cost of the plant.*—The cost of items of plant equipment was determined unit by unit as carefully as possible from a consideration of the cost of materials, probable cost of their fabrication, and cost of instruments, motors, &c., all at rates prevailing at the time of writing.

Digester residue disposal costs were estimated on the basis of provision of equipment for removal of half the volume of residue as water from each batch and the carting by motor truck of the remainder during one shift.

The cost of the plant grouped into units was as follows:—

Digestion unit	£1,300
Distillation unit	900
Hull storage and handling ..	3,300
Tanks and fittings	1,100
Residue handling equipment ..	1,450
Pipes and fittings	700
Erection and installation ..	1,000
Instruments	550
Design costs	500
Total	£10,800

A figure of £11,000 was taken as the plant cost.

(b) *Cost of Production.*

Details of the items contributing to the production cost are shown in Table 19. The cost of production was first determined on the basis of the plant being an adjunct to an existing plant with adequate excess boiler capacity and then the additional cost of boiler facilities was determined for its operation as an individual plant.

TABLE 19.—DETAILS OF COST OF FURFURAL PRODUCTION.*

Item.	Quantity Per Day.	Bulk Cost.	Cost/Unit (pence).	Units/lb. Furfural.	Cost/lb. Furfural (pence).	Percentage of Total Cost Including Boiler.
Oat-hulls	7 21 tons	£1 14s. per ton	0 183 per lb.	10 11	1 853	16·7
Steam	58,200 lb.	5s. per 1,000 lb.	0 06 per lb.	36 35	2 181	19·6
Water-cooling	44,300 gal.	6d. per 1,000 gal.	0 006 per gal.	27·70	0 166	1·5
Water-charge	2,400 gal.	6d. per 1,000 gal.	0 006 per gal.	1 50	0 009	0 1
Sulphuric acid (94 per cent.)	303 lb.	£9 16s. 6d. per ton	1 05 per lb.	0 189	0 198	1 8
Electric power	161 kw. hr.	0 98d. per kw. hr.	0 98 per kw. hr.	0 101	0 099	0 9
Labour—						
Process workers	6 (2 per shift)	£6 10s. per week			1 170	
Labourers	6 (2 per shift)	£5 10s. per week			0 990	
Truck driver	1	£6 10s. per week			0 195	28 4
Fitters	2	£7 10s. per week			0 450	
Technical control	1	£12 per week			0 360	
Depreciation on £11,000 at 10 per cent.	£4 4				0 660	
Interest on £11,000 at 5 per cent.	£2 2				0 330	14 9
Maintenance on £11,000 at 10 per cent.	£4 4				0 660	
Running costs of waste disposal truck	65 miles	1s. per mile			0 488	4 4
Containers—200 returnable drums, life 2 years	3 (44 gal.)	£3 each			0 180	1 6
Total without boiler					9 989	89 9
Boiler installation—depreciation on £6,000 at 10 per cent.	£2 4				0 360	3 2
Interest on £6,000 at 5 per cent.	£1 2				0 180	1 6
Boiler attendants	3 (1 per shift)	£6 10s. per week			0 585	5 3
Boiler costs					1 125	10 1
Total with boiler					11 114	100

* On a basis of 200 short tons of furfural per annum.

The cost figures and material and energy requirements per pound of furfural which are shown in Table 19 form a suitable basis on which the cost of furfural production may be calculated for a wide range of conditions.

From these results it can be seen that the cost of producing furfural in Australia from oat-hulls would be unlikely to exceed 11d. per pound, exclusive of overhead charges, as compared with the present price of approximately 1s. 4d. for imported furfural in ton lots, duty paid, on wharf in Sydney.

6. General Conclusions

The pilot plant investigation of the production of furfural from oat-hulls and maize-cores has shown that these Australian raw materials are satisfactory, has enabled the optimum production conditions to be defined and has supplied data necessary for the design of larger furfural digestion equipment.

The study of the recovery of furfural from its aqueous solutions by distillation has shown the McCabe-Thiele method to be suitable for the design of furfural distillation equipment and has enabled a choice to be made of equipment required for this separation.

The estimate made of the cost of furfural production from oat-hulls has shown Australian production of this substance on a scale of 200 tons per annum to be economically feasible provided the cost of raw material and overhead charges are not excessive.

7. Acknowledgments

8. References

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The Yield of Furfural from Pentosans

By B. W. Wilson, M.Sc.*

Summary.

The reasons for the low yield obtained in industrial production of furfural have been investigated.

A study of the hydrolysis and dehydration reactions involved showed that the limiting factor was the side reactions which occur in the dehydration of pentoses to furfural.

The yields obtained in both single and two stage digestion processes were compared and the relative merits of the two processes are discussed.

1. Introduction

The low yield of furfural obtained from naturally occurring pentosans has been one of the main problems of furfural production since it was first undertaken on an industrial scale.

In developing a process suitable for producing furfural in Australia† a stage was reached when the yield could not be improved beyond 50 per cent. without introducing extra equipment or radically altering the technique of operation. It seemed apparent that further improvement, if it were possible at all, would depend on a more thorough understanding of the reactions which influence the yield. The present paper describes a study of these reactions.

The progress of the two main reactions, the hydrolysis of pentosans to pentoses and the dehydration of pentoses to furfural, which occur almost simultaneously, could not be followed directly in the digestion process but, because the first reaction was more rapid, it was possible to study each in turn and to determine the nature of the loss in yield.

Although it was found impractical to improve the yield of furfural, the results of this study demonstrated the mechanism of the reactions concerned and led to a better appreciation of the factors involved in the production of furfural. In addition it was found that the lignocellulose produced by low temperature hydrolysis of pentosans in oat-hulls might be useful as a filler for plastics. Tests on this material have been described in an accompanying paper‡.

2. The Hydrolysis of Pentosans

(i) *Introduction.*

Previous study of the hydrolysis of pentosans has been concerned mainly with technical processes such as the preparation of furfural, xylose, and low pentosan pulps (1). Information on the conditions and mechanism of reaction, and on yields, is limited when compared with the knowledge that has been accumulated on wood saccharification, which is a similar process. In this case Saeman (2) and others have demonstrated that two consecutive reactions, the

hydrolysis of cellulose and the decomposition of the resulting hexose sugars, were the main reactions determining the yield of sugars and that maximum yield of hexoses depended on the selection of conditions such that the hydrolysis proceeded at a maximum and decomposition at a minimum rate.

Although a similar mechanism was expected to apply in the production of pentoses by hydrolysis of pentosans, a quantitative treatment of the reaction was faced with a number of difficulties. The pentosans are not uniform materials and the term pentosan covers a wide range of substances (3) some of which hydrolyse more readily than cellulose and some of which are more resistant than cellulose. In addition, the nature and quantity of these materials cannot be precisely estimated and they can only be assessed in terms of the furfural yielded when the material is distilled with 12 per cent. hydrochloric acid under controlled conditions (5). The products of hydrolysis of pentosans which are referred to as " pentoses " in this paper, have not yet been clearly defined and it is not possible to specify the constituents that yield furfural. The present study of the hydrolysis of pentosans was limited to determining the extent the two reactions described above controlled the yield of pentoses, with conditions ranging from those used by Dunning and Lathrop (4) who obtained 90-95 per cent. yields of pentoses from maize cores, to those used in the furfural digestion referred to in the accompanying paper*.

(ii) *Apparatus and Method.*

The small furfural digester used for the process study described in the accompanying paper† was used for the experiments on the hydrolysis of pentosans in oat-hulls.

The operating procedure was as follows: After preheating with live steam, the digester was loaded with 30 lb. of oat-hulls and 100 lb. dilute sulphuric acid, made up to the required strength with commercial sulphuric acid (93 per cent. H_2SO_4), and the charge heated with live steam until the working pressure was reached (5-10 min.). It may be assumed that little hydrolysis occurs during this period and the time of reaction was measured from the instant the working pressure was reached when the stirrer was started. During hydrolysis the steam jacket pressure was adjusted to a value just sufficient to maintain working pressure while live steam was passed into the hydrolysis mixture and vapour containing small quantities of furfural was removed and condensed. At the end of the run the steam pressure was reduced and the contents of the digester discharged into a stainless steel hydro-extractor with an 18 in. diameter basket filled with stainless steel gauze. The solid residue was washed in the basket with 5-6 gal. of water and the combined washings and hydrolysis solution weighed and sampled. The solid residue was also weighed, mixed, and sampled. The potential furfural content of the pentose solution and the unchanged pentosan in the residue were estimated by the usual methods (4, 5), and yields were calculated on the original potential furfural content of the oat-hulls.

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* This Journal p. 225.
† Ibid. p. 227.

(iii) *Results and Discussion.*

The results of hydrolysis experiments at 100°, 135°, 150°, and 180° C. are shown in the accompanying graphs (Figs. 1, 2, 3, 4). The time allowed for hydrolysis and the acid concentration were selected in each case so that only conditions close to those for optimum yields of pentoses were studied. Fig. 1 shows the yields obtained with conditions similar to those used by Dunning and Lathrop (4) for the hydrolysis of maize-cores. With acid concentrations up to 4·0 per cent. the yield of pentoses was almost exclusively limited by the amount of pentosans remaining unhydrolysed. With higher acid concentration the remaining 10-15 per cent. of the pentosans resisted hydrolysis and the yield decreased because of decomposition reactions caused by the increased acid concentration. In order to use more vigorous conditions to reduce the amount of unchanged pentosans and at the same time to keep the decomposition of pentoses at a low value, a two-stage hydrolysis was planned on the results shown in Fig. 1. In the first stage, 2 per cent. acid was used and this was followed by a second treatment with 6 per cent. acid. The yield of 83·4 per cent. pentoses and 9·3 per cent. unchanged pentosan was a slight improvement on the best yields obtained at 100° C. with a single stage.
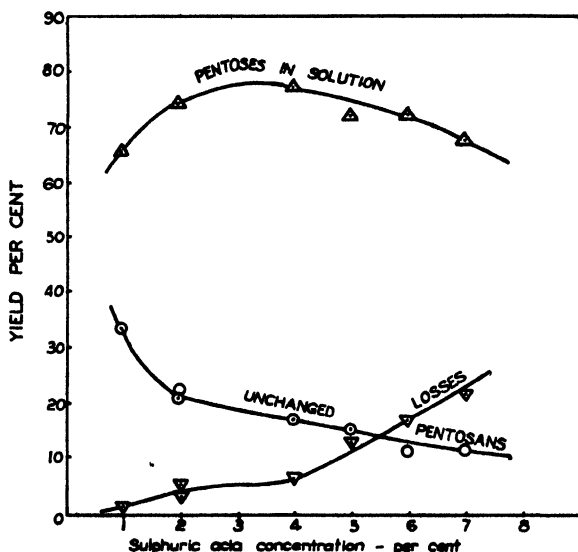
Fig 1.—Hydrolysis for 2 hr. at 100° C.

The advantages of a two-stage hydrolysis were discounted by results of further single-stage experiments at 135° C. where it was found that the increase in temperature together with a lower acid concentration gave a yield of 83·5 per cent. pentoses and 9·0 per cent. unchanged pentosans (Fig. 2).

With a further increase in the temperature of hydrolysis (Figs. 3 and 4), the maximum yield decreased because the decomposition of pentoses increased at a greater rate than the rate of hydrolysis. The increased rate of decomposition was evident from the increased amount of furfural actually formed which has been shown as a separate source of loss in yield of pentoses in Figs. 2, 3, and 4.

Although the hydrolysis of pentosans to pentoses was complicated by the formation of furfural, the above experiments provided conclusive evidence that the factors limiting the yield were similar to those encountered in wood saccharification. Up to 75 per cent. of the pentosans hydrolysed readily with very small losses due to decomposition reactions but the remainder showed so much resistance that the more vigorous conditions required led to increased decomposition which was at least equal to the additional pentosans hydrolysed. From
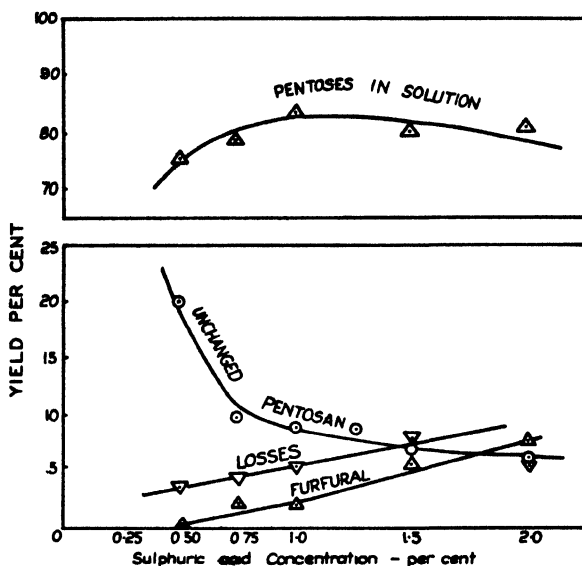
FIG. 2.—Hydrolysis for 2 hr. at 135° C.
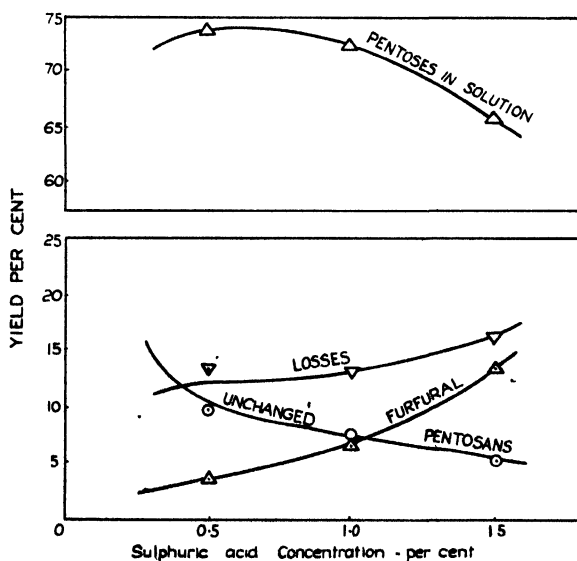
FIG. 3.—Hydrolysis for 1 hr. at 150° C.

the point of view of obtaining a maximum yield of pentoses from oat-hulls, hydrolysis at 135° C. was best. At higher temperatures the yield was reduced by dehydration of some of the pentoses to furfural but this would not matter in a commercial process for producing furfural.
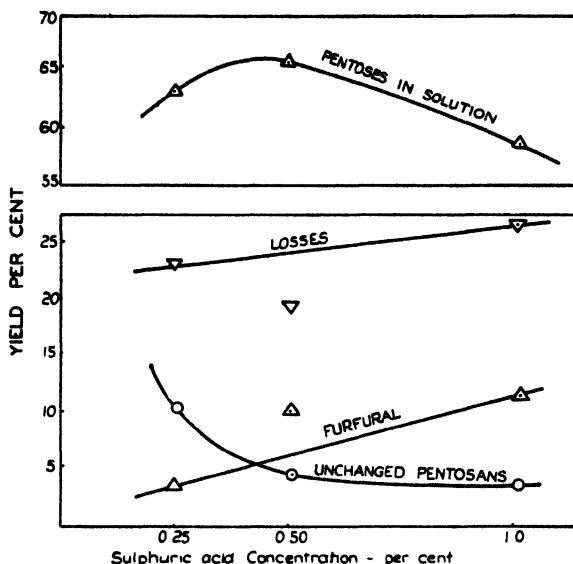
FIG. 4.—Hydrolysis for 15 min. at 180° C.

3. The Dehydration of Pentoses to Furfural

(i) *Introduction.*

From the above study of the hydrolysis of pentosans it was apparent that the main loss in yield in furfural processes was associated with the dehydration of pentoses to furfural. Apart from one claim of a quantitative yield (6), this confirmed the results of previous workers which showed that the yield of furfural obtained from the pentoses formed by hydrolysis of pentosans or from pure pentoses such as xylose seldom exceeded 50 per cent. theoretical (1, 4). Two reactions leading to the destruction of furfural or furfural-yielding substances have been suggested as being responsible for the loss in yield. The readiness with which furfural polymerizes has been regarded for some time as a potential source of loss which can be partly overcome by removing furfural from the reaction by means of steam distillation but, in view of experiments on the stability of furfural (7, 8) under the conditions of reaction and recent work on the decomposition of hexose and pentose sugars (2), it was apparent that some at least of the loss was caused by pentoses undergoing reactions to yield unreactive caramel-like substances.

The present investigation aimed at determining the effect of the conditions of reaction on the yield of furfural and the extent to which the destructive reactions affected the yield. Preliminary experiments in the digester with pentose solutions obtained from the hydrolysis of

pentosans showed that yields of furfural up to 50 per cent. were possible, but as this equipment was unsuitable for detailed study further work was carried out in a laboratory autoclave.

(ii) *Apparatus and Method.*

The autoclave was constructed from a 6-in. length of 3-in. diameter copper tube, with a dished cover of $\frac{1}{4}$-in. copper sheet brazed to one end. The other end which was flanged was closed by a bronze cover secured by means of two steel backing flanges with six bolts. A steam pressure gauge and copper inlet and outlet tubes fitted with small nickel-seated valves were fitted to the cover. Heat was supplied by means of two electric furnaces. One was fitted with two 1,650-watt elements and was used to provide rapid initial heating, while the other which had a single 1,650-watt element controlled by a variable transformer was used to maintain the autoclave at working temperature.

For each experiment 250 ml. of pentose solution containing a known concentration of sulphuric acid was measured into the autoclave, the cover bolted in place, and the autoclave suspended inside the first electric furnace which had been preheated for 20 minutes. When the liquid boiled—after about 2 minutes—the outlet valve was closed and pressure allowed to build up to 150 lb./sq.in. in 2-3 minutes. The autoclave was removed quickly and placed in the second furnace where the pressure was controlled at 130 lb./sq.in. by means of the transformer.

At the end of the run which was timed from the moment the pressure reached 150lb./sq.in., the autoclave was plunged into cold water, and the pressure reduced to atmospheric in about 30 seconds. When the autoclave was opened, the solution which contained a dark-brown tarry suspension with an accumulation of black solids at the surface of the liquid, was filtered and sampled One portion was steam distilled to obtain free furfural while another was distilled with 12 per cent. hydrochloric acid to obtain the total furfural content including that from unchanged pentoses. The furfural loss due to unchanged pentose was obtained from these results by difference.

The pentose solution used in these experiments was obtained by hydrolysis of oat-hulls with 1 per cent. sulphuric acid for 2 hours at 30 lb./sq.in. with a liquid/solid ratio of 3·33. Under these conditions 75 per cent. of the pentosans were dissolved leaving a light-coloured residue which was later tested as a filler for plastics*. The pentose solution contained 5·10 per cent. potential furfural and only a negligible quantity of free furfural. The acid concentration was adjusted by the addition of sulphuric acid or caustic soda to give the required pH which was measured by means of a glass electrode.

(iii) *Results and Discussion.*

(a) *The yield of . furfural from pentoses.*—The effect of acid concentration and reaction time on the yield of furfural from pentoses at 180° C. has been shown in Fig. 5 where results have been plotted as yield of furfural against time for different acid concentrations. The shape of the curves indicated that two consecutive reactions were taking place with the second slower than the first. However, as the

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* This Journal p. 272.

maximum yields were obtained after longer time intervals with no substantial increase in yield when the acid concentration was decreased, and as the second reaction, i.e. the decomposition of furfural, then became too slow to account for the low yield of furfural, some other reaction must have been responsible. The decomposition of pentoses to tarry materials simultaneously with the dehydration to furfural appeared to be the most likely reaction, and this was confirmed by experiments with pure xylose which have been described in a following section.
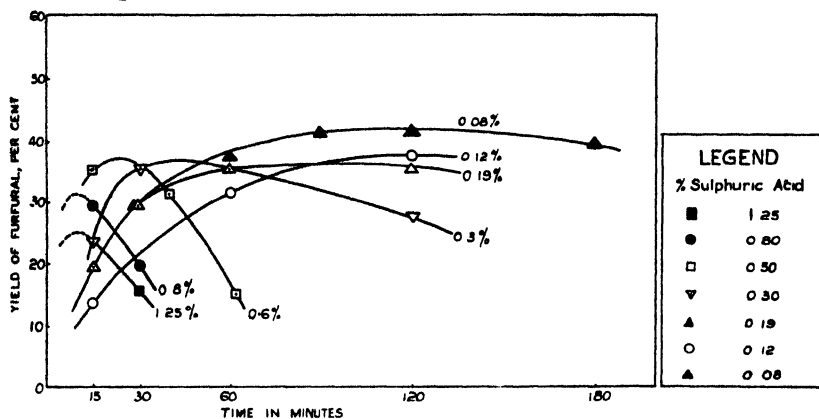
FIG. 5.—Yield of furfural from dehydration of pentoses at 180° C.

Apart from indicating the cause of loss of potential furfural, the results were significant from a practical point of view. The range of acid concentration from 0·80 to 1·25 per cent. represented conditions under which a continuous process might be operated for the manufacture of furfural from pentoses. Over this range the reactions proceeded very rapidly and it was not possible to distinguish the maximum yield. Between 0·30 and 0·80 per cent. conditions were similar to those in the single-stage furfural digestion and indicated that the furfural-forming reactions were practically complete in 15-60 minutes. The remaining curves from 0·08 to 0·30 per cent. showed that yields up to 40 per cent. theoretical could be obtained in a digestion process in which furfural was not removed by distillation during digestion.

(b) *Unchanged pentoses.*—The effect of variation of the conditions of dehydration on the yield of unchanged pentoses has been shown in Fig. 6. The curves confirmed the rapidity of the dehydration in the early stages and showed that loss in yield was not due to incomplete conversion of pentoses to furfural. The effect of variation of acid concentration on the rate of dehydration appeared to be small over the range 0·30 to 0·08 per cent. sulphuric acid. Comparison with the furfural yield curves also made it evident that a reaction, probably the decomposition of pentose to non-furfural yielding substances, was taking place simultaneously with the dehydration reaction.

(c) *The formation of solids.*—The variation of the amount of solids formed when pentose solutions were treated in an autoclave at 180° C. with varying times and acid concentrations has been shown in Fig. 7.

The yield of solids could not be related directly to the yield of furfural and unchanged pentoses because it was impossible to express the amount of solids in terms of an equivalent amount of furfural, but the similar trends of the curves in the earlier stages of the reaction and similar variations with different acid concentrations (compare Figs. 6 and 7) indicated that the yield of solids was related to the furfural forming reactions and not entirely due to the decomposition of gums or other substances extracted from the oat-hulls.
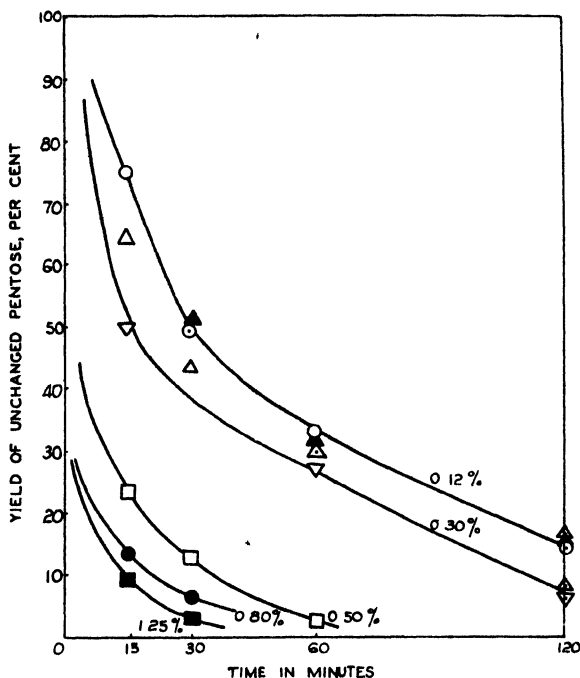
Fig 6   Yield of unchanged pentoses from dehydration of pentoses at 180° C

The main value of these results was that they showed that provision for removing solid products of reaction must be an important feature of any process, either batch or continuous, which is designed for recovering furfural from pentose solutions. Under the conditions for a maximum yield of furfural which can be ascertained from Fig. 5, the amount of solid formed—which can be determined from Fig. 7— was 30-40 per cent. by weight of the potential furfural in the pentose solution.

(d) *Comparison of the dehydration of crude pentoses and pure xylose.*—The extent that the formation of furfural from pentoses from the hydrolysis of pentosans differed from the formation of furfural from pure xylose was determined by a series of experiments with solutions of pure xylose with a potential furfural content similar to the pentose solution used in experiments described previously. 200 ml. aliquots of solution were heated at 180° C. for varying intervals of time with 0·08 per cent. sulphuric acid. The resulting

yields of furfural and unchanged xylose have been shown in Fig. 9 where comparison has been made with the yields obtained under the same conditions with a pentose solution obtained by hydrolysis.
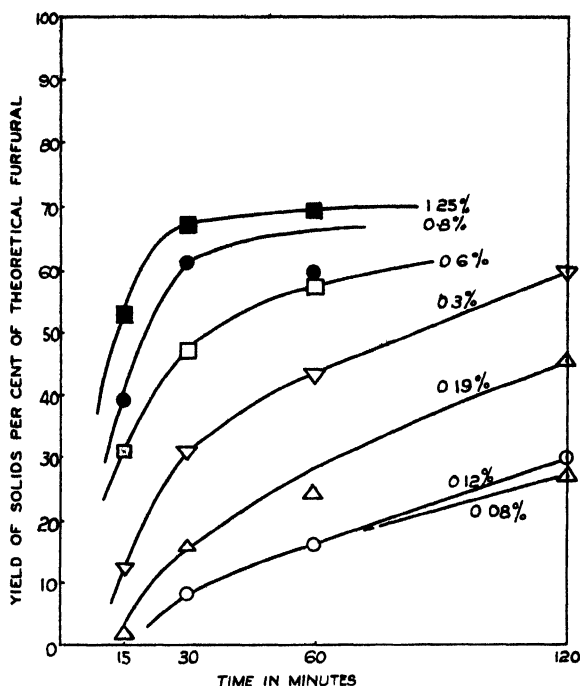
FIG 7.—Yield of solids from dehydration of pentoses at 180° C

For xylose, the rate of decomposition and the rate of formation of furfural were slower than for pentoses, indicating that the pentose solution contained furfural-yielding substances which reacted more readily than xylose and that experiments with xylose were of limited value in predicting yields from pentosans.

(e) *The decomposition of xylose and furfural.*—The experiments with xylose were followed by a study of the decomposition of pure furfural under the same conditions. The results have been shown in Fig. 8 where the percentage of residual furfural has been plotted on a logarithmic scale against time and compared with a similar plot of the decomposition of xylose. The graphs showed that both reactions were of the first order with velocity constants of 0·0012 min.$^{-1}$ for furfural and 0·015 min.$^{-1}$ for xylose.

The relatively low value of the velocity constant for furfural and the results of previous study showed that the decomposition of furfural after it had been formed could not account for all the loss in yield and that the decomposition of xylose to tarry products during its dehydration to furfural must have been mainly responsible.
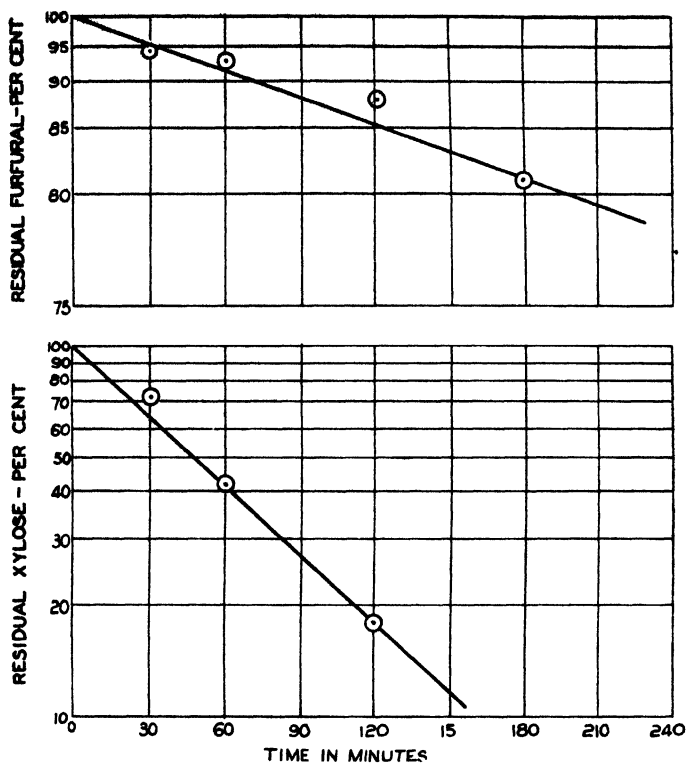
FIG 8 —Decomposition of xylose and furfural in 0 08 per cent sulphuric acid at 180° C

(f) Kinetics of the dehydration of xylose.—The data in the previous section indicated that the dehydration of xylose involved two simultaneous and one consecutive reaction which could be represented as follows:
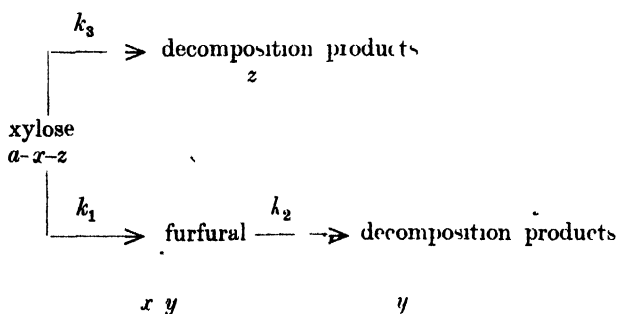
The kinetics of these reactions were derived as follows: If after time t, x mol. of furfural, z mol. of xylose decomposition products, and y mol. of furfural decomposition products have been formed from

a mol. of xylose and if k_1, k_3, and k_2 were the respective first order reaction constants, the net furfural was $x—y$ mol. and the quantities of each material could be represented by the following formulas:—

The amount of unchanged pentose $= ae^{-(k_1 + k_3)t}$

The amount of furfural formed, $x = \dfrac{k_1a}{k_1 + k_3}\left(1 - e^{-k_1t}\right)$

The amount of xylose decomposition products,

$$z = \dfrac{k_3a\left(1 - e^{-k_3t}\right)}{k_1 + k_3}.$$

The amount of furfural decomposition products,

$$y = \dfrac{\dfrac{k_1k_2a\left(1 - e^{-k_1t}\right)}{k_1 + k_3} - \dfrac{k_1^2a\left(1 - e^{-k_2t}\right)}{k_1 + k_3}}{k_2 - k_1}$$

The amount of net furfural

$$x - y = \dfrac{k_1^2a\left(e^{-k_1t} - e^{-k_2t}\right)}{(k_1 + k_3)(k_2 - k_1)}$$

These formulae which were based on the formulas given by Saeman for the saccharification of cellulose, enabled k_1 to be calculated by substituting for $k_1 + k_3$ and k_2 $(0 \cdot 015$ and $0 \cdot 0012$ min.$^{-1})$ and a known value for the net furfural at a given time (38 per cent. in 60 mins.). With this value $(0 \cdot 0117$ min.$^{-1})$ it was possible to calculate the maximum yield of net furfural by means of the expression:

$$x_{max} = \dfrac{k_1}{k_1 + k_3}\left(\dfrac{k_2}{k_1}\right)^{\frac{k_2}{k_1 - k_2}}$$

For the decomposition of xylose at 180° C. and $0 \cdot 08$ per cent. catalyst concentration the maximum yield of furfural was 60 per cent., which might be increased to 78 per cent. if the decomposition of furfural was prevented by distilling off the furfural during the reaction.

In Fig. 9 the yield of furfural has been plotted for a value of $k_1 = 0 \cdot 0117$. The calculated curves agree closely with the observed results in the early stages of the reaction but diverged slightly as

the optimum yield was approached. No matter how close an agreement might be obtained, however, it cannot be claimed that this represents a verification of the above theory until a value for k_1 can be obtained by an alternative method.
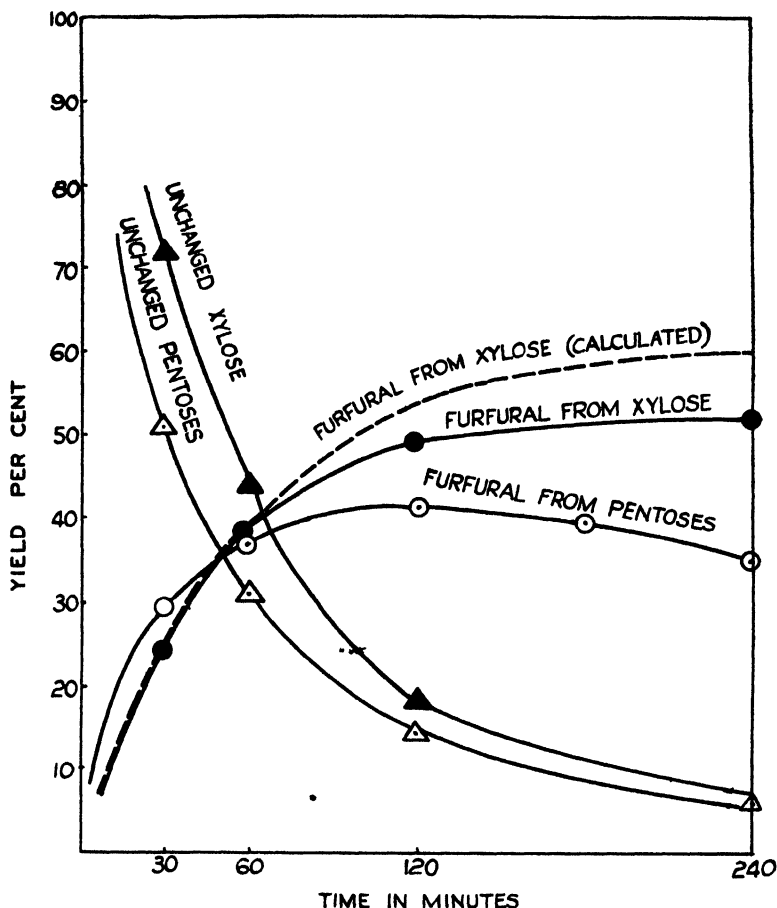
FIG. 9.—Comparison of the dehydration of pure xylose with the dehydration of the pentoses from pentosans.

4. The Utilization of Pentosan-containing Materials

The production of furfural on an industrial scale has been achieved by two different techniques which can be distinguished by the use made of the lignocellulose which accompanies the pentosans. When the demand for furfural is large, the raw materials cheap, and the lignocellulose can be regarded as a waste, a single-stage digestion process provides the most economic method of furfural production. When a suitable outlet for the lignocellulose can be developed, as for example in the production of alcohol (4) or paper pulp (1), a two-stage process in which the lignocellulose is recovered after a mild hydrolysis of the pentosans and the furfural recovered by treatment

of the pentose solution, provides an alternative method. This enables full use to be made of the raw material, and permits the utilization of materials that are too low in pentosan for a single-stage process.

· The results of tests of lignocellulose wastes from furfural processes as fillers for plastics*, showed that a form of lignocellulose with properties similar to wood flour could be produced by the hydrolysis of oat-hulls under mild conditions. In adidtion, a moderate yield of furfural can be obtained in this process by autoclaving the hydrolysis liquors at 180°C.

The lignocellulose filler was prepared by digesting 50 lb. batches of oat-hulls with 1 per cent. sulphuric acid for two hours at 30 lb./sq.in. digester pressure and a liquid/solid ratio of 3·33 in the small digester
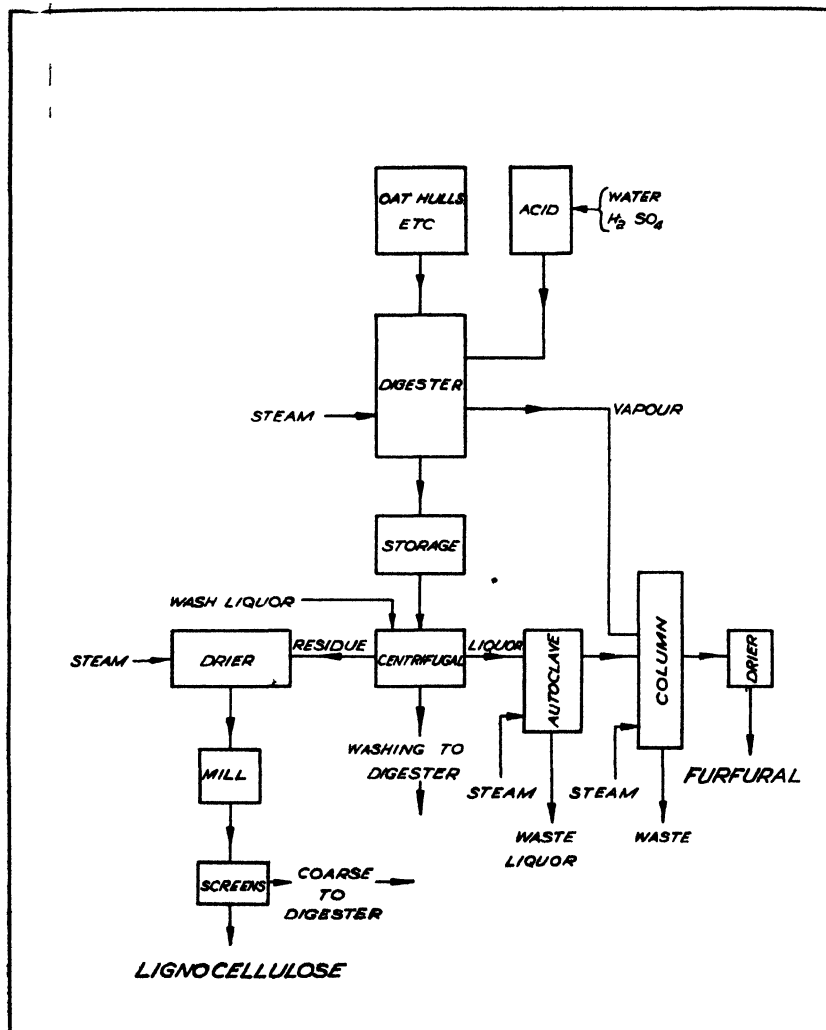
FIG. 10.—A process for complete utilization of pentosan—containing material.

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used in earlier experiments. The lignocellulose was filtered, washed, dried, and passed through a disintegrator to break up the cake after drying. 85 per cent. of the dry lignocellulose passed through a 50-mesh wire sieve giving an over-all yield of 44 lb. of air-dry lignocellulose per 100 lb. of oat-hulls. The average yields of pentose and furfural were as follows:

Pentose in solution	.	73·5 per cent.
Unchanged pentosans	..	18·5 per cent.
Actual furfural ..	..	2·7 per cent.
Unaccounted ..	..	4·3 per cent.

The relatively large amount of unchanged pentosans was caused by the choice of conditions which had the least damaging effect on the lignocellulose. The pentoses were dehydrated to furfural with a yield of 50 per cent. giving an over-all yield of 7·5 lb. of furfural per 100 lb. of oat-hulls.

In a large scale two-stage furfural process as shown in Fig. 10, 880 lb. of lignocellulose and 150 lb. of furfural would be produced from 2,000 lb. of oat-hulls compared with 200 lb. of furfural obtained from the same quantity in a single-stage process.

If the lignocellulose produced from oat-hulls was acceptable as a substitute for wood flour, the two-stage process would merit further development in spite of the lower yield of furfural because the demand for fillers in the plastics industry is considerably greater than the demand for furfural.

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Lignocellulose Residues as Fillers for Phenolic Plastics

By L. K. Dalton, A.S.T.C.*

Summary.

Two lignocellulose residues, obtained from the high and low-temperature processes for the preparation of furfural, have been tested as fillers for phenolic plastics. They were compared with wood-flour for their effect on the tensile and impact strengths, plastic flow, and the water resistance of the plastic.

1. Introduction

The lignocellulose by-product from the production of furfural by the digestion of oat-hulls has been examined as a filler for plastics. Two lignocellulose residues were tested. The one was a by-product from the preparation of pentoses from oat-hulls by digestion at 135°C. with 1 per cent. sulphuric acid for two hours, as described by Wilson.† It is subsequently referred to as lignocellulose I. The other was a by-product from the digestion of oat-hulls at 180°C. with 1·5 per cent. sulphuric acid with simultaneous distillation to remove furfural, as described by Brown, Symons, and Wilson.‡ It is referred to as lignocellulose II. The first was light-brown, contained 28 per cent. lignin,§ and it had been ground to — 50 mesh. The second was dark-brown, contained 48 per cent. lignin,§ and it had been ground to — 72 mesh. Both residues had been washed free from acid.

They were compared with a commercial wood-flour in a two-stage phenolic plastic composition. The wood-flour was slightly coloured and had been ground to — 50 mesh. Moulded specimens were prepared for the determination of the ultimate tensile strength, the Izod impact strength, and the water absorption. Cup-flow times were determined.

2. Preparation of Moulding Powders

Duplicate batches of moulding powders were prepared for each filler according to the formula given below, and two series of powders were prepared using different methods of mixing.

FORMULA OF MOULDING POWDER.	g.
Novolak resin	400
Hexamine	48
Calcium stearate	8
Lime	8
Filler	400

* An officer of the Division of Industrial Chemistry.
† This *Journal*, p. 258.
‡ This *Journal*, p. 225.
§ Determined as the insoluble in 72 per cent. sulphuric acid. See Ott, E.— "Cellulose and Cellulose Derivatives." 1st. Ed., p. 141. (New York: Interscience Publishers, 1943.)

The Novolak resin was prepared from phenol (1·0 g. mol.) with formaldehyde (0·8 g. mol.) using 0·5 per cent. of the total weight of oxalic acid as catalyst, and with sufficient water to yield a product containing 60 per cent. resin. The mixture was heated in an autoclave for 120 minutes under a pressure of 10 lb./sq. in., after which the excess phenol was steam-distilled under vacuum and the resin finally dehydrated by vacuum distillation. The resin was cooled in shallow trays and then ground in a hammer mill to — 100 mesh. The softening point of the resin was 90°C.

The powders were mixed either for 20 hours in porcelain pebble mills or for 3 hours in half-gallon drums, fitted with three one-inch baffles.

After mixing, the powders were hot-milled for 9 minutes on rolls, one of which was heated with steam at a pressure of 25 lb./sq. in. and the other cooled by running water. The sheets from the rolls were crushed and then ground to a granular powder in a Christy and Norris mill.

3. Moulding

For the determination of ultimate tensile strength and Izod impact strength, test specimens were moulded according to B.S.S. 771—1938, and for water absorption according to A.S.T.M. 570—1942. The cup-flow time was determined according to B.S.S. 771—1938. The conditions of moulding are given in Table 1.

TABLE 1.—CONDITIONS OF MOULDING.

Test.	Number of Specimens.	Pre-heating		Moulding			Period Before Testing
		Time.	Temp.	Time.	Temp.	Pressure.	
		min.	°C.	min.	°C.	lb./sq. in.	days.
Tensile and impact ..	8	15	105	15	160–5	3,400	7
Water absorption ..	3	15	105	6	160–5	3,400	2
Cup flow	2	..	..	..	163	Total pressure, 10 tons	..

4. Results

The results of the tests on the moulded specimens are given in Table 2.

The properties of the moulded plastics depended to some extent on whether the moulding powder had been mixed in the drum or in the ball-mill. It was thought that the lignocellulose fibres, embrittled by the digestion, would be better preserved by using the drum. However, an increase in tensile strength was obtained only for the plastic filled with lignocellulose I and the impact strengths were generally lower. The cup-flow times were lower and the water

absorption higher when the drum was used. Much more extensive testing would be required to determine the effect of this and other variables, but these results are sufficient to give a preliminary estimate of the relative value of these materials as fillers.

TABLE 2.

Test	Ball-Mill			Revolving Drum		
	Wood-flour	L.C. I	L.C. II	Wood-flour.	L.C. I.	L.C. II
Izod Impact Strength* (ft.lb./½ in notch)—						
Batch 1	0·21	0·23	0·18	0·18	0·24	0·15
,, 2	0·22	0·23	0·17	0·18	0·20	0·14
Mean	0·22	0·23	0·18	0·18	0·22	0·15
Ultimate Tensile Strength* (lb./sq. in)—						
Batch 1	5,720	5,130	3,260	5,400	5,630	3,010
,. 2	6,040	4,730	3,820	5,580	5,860	1,880
Mean	5,880	4,930	3,540	5,490	5,750	2,450
Water Absorption (mg)† 24 hrs. immersion—						
Batch 1	36	25	17	33	32	18
., 2	35	24	17	34	30	18
Mean	36	25	17	34	31	18
Water Absorption (mg.)† 7 days' immersion—						
Batch 1	112	72	46	112	102	52
,, 2	106	69	48	112	92	51
Mean	109	71	47	112	97	52
Cup Flow Time (secs)‡—						
Batch 1	16	15	16	11	12	12
,, 2	15	16	16	11	12	12
Mean	16	16	16	11	12	12

* Mean values for 8 specimens from each batch.
† Mean values for 3 specimens from each batch.
‡ Mean values for 2 tests on each batch.

The wood-flour filled plastic used for comparison is classified bv the B.S.S.771—1938 as the general type, which has an ultimate tensile strength of 5,000 lb./sq. in. and an Izod impact strength of 0·11 ft.lb./½-in. notch.

When used as a filler lignocellulose I. produced tensile and impact strengths almost the same as obtained with wood-flour filler, and it gave an appreciably lower water absorption. Lignocellulose II. gave a much lower tensile strength and a slightly lower impact strength, but a considerably better, lower, water absorption.

The change of filler had no effect on the flow time of the plastic. As the plastics containing lignocellulose II. did not have lower cup-flow times than those containing wood-flour, it can be concluded that the lignin in this filler does not contribute to the flow of the

plastic and therefore cannot be used to extend the resin. This was confirmed with a powder prepared with lignocellulose II. replacing in part both the wood-flour and the resin. A h rd-flowing, low-strength plastic was obtained.

As no dye or pigment was used in the plas ic the base colours could be compared. Although lignocellulose I. was darker than the wood-flour, it gave a lighter coloured moulding. However, wood-flour is obtainable with less colour than the one used. Lignocellulose II. gave black mouldings

5. Conclusions

The lignocellulose residue obtained by acid digestion at 135°C. could be used to replace wood-flour as a filler in phenolic plastics. The development of this process for the production of furfural and lignocellulose for use as a filler would require further study of the reaction to determine the conditions which give a filler with the best properties while obtaining an economic yield of furfural. A use for the residue might be found in the manufacture of linoleum and building boards.

The lignocellulose residue obtained by acid digestion at 180°C. is unsuitable as a filler for phenolic plastics, although it does improve their water resistance. Despite its high lignin content this residue is also unsuitable as a resin extender, and for this purpose it would have to compete with the vast quantities of pure lignin which the paper industry is capable of producing.

Molecular Weight Distribution Curves of Various Holocellulose Fractions

By *Beulah Brims, B.Sc.**

Summary.

Four residual fractions were prepared from *Eucalyptus regnans* F. v. M. holocellulose by the use of boiling water and sodium hydroxide solutions of three different strengths. Molecular weight distribution curves were determined for the holocellulose and its fractions. There are indications that, from the point of view of molecular weight distribution, the carbohydrates of *E. regnans* holocellulose exist in two colonies, i.e., the holocellulose represents a rather heterogeneous material consisting chiefly of portions of very low and very high molecular weight, with only a small percentage having intermediate values. Treatment with boiling water caused a slight increase in the size of the first holocellulose colony, the second being correspondingly reduced, while treatment with alkali as weak as 0·2 per cent. caused a very marked increase in the size of the first colony, the second being so reduced that it represented only a very small percentage of the original holocellulose. Similar results were obtained by treatment with stronger alkali solutions. Concurrent with this decrease in chain length, removal of xylan occurred. Possible reasons for this decrease are discussed.

1. Introduction

In early studies on the hemicelluloses of wood, the method of preparation of the hemicellulose fractions involved the extraction of wood with alkali. Later, procedures were developed for the almost complete delignification of cellulosic materials, the residue containing all, or nearly all, the polysaccharides of the cell wall. The " skelettsubstanzen " of Schmidt and the " holocellulose " of van Beckum and Ritter are examples of such residues which may be taken as representing the cellulose, cellulosans, and polyuronide hemicelluloses. Using holocellulose as a starting material, several workers have subsequently obtained hemicellulosic fractions by treatment with alkali solutions, and have analysed these for nitrogen, acetyl, methoxyl, xylan, and glucosan contents. Little attention has been paid, however, to the distribution of chain length in the residues obtained, or in the material extracted, by treatment of holocellulose with alkali.

Following on an investigation of Ekenstam's (1942) method for measuring the polymolecularity of pulps, it was thought that some interesting results might be revealed by determining the molecular weight distribution curves of some alkali fractions prepared from *E. regnans* holocellulose.

2. Experimental

The material used was the 60-80 mesh fraction from shavings of *E. regnans* which had been ground in a Wiley mill. The wood meal was extracted with distilled water, and this treatment was followed by a cold, then hot, alcohol extraction.

* An officer of the Division of Forest Products.

The total carbohydrate fraction was determined by alternate treatments with chlorine water and cold ammoniacal alcohol (10 ml. ammonia of S.G. 0·880 and 90 ml. 99 per cent. alcohol). This method was originally used by Kerr and Bailey (1934) for the complete removal of lignin from wood sections, and has been modified in this laboratory (unpublished work) to be applicable to analytical studies. Seven chlorination cycles were employed, and the product, when examined, was found to contain all the furfural-yielding groups that were present in the original wood. The lignin value was found to be 1·5 per cent., and, since this was high, an investigation was carried out with the object of determining the number of chlorination cycles which were necessary for the lowest lignin value, while still retaining the furfural-yielding groups. It was established that eight cycles were necessary. A bulk sample of holocellulose was then prepared, having the same pentosan and with the same yield as the material obtained on the crucible scale with eight chlorination cycles. It was found possible to prepare such samples in amounts of 50 g. at a time.

The bulk sample of holocellulose represented 81·3 per cent. of the moisture-free weight of the extracted wood, and gave a furfural yield, calculated as xylan, of 21·9 per cent. From it, three residual fractions were prepared by treatment with 0·2 per cent., 5 per cent., and 10 per cent. sodium hydroxide respectively.

In the preparation of the 0·2 per cent. sodium hydroxide fraction, two samples, each of 7·5 g. of air-dry holocellulose, were allowed to stand for two hours, with occasional shaking, in contact with 375 ml. of aqueous sodium hydroxide containing 0·75 g. of sodium hydroxide. The residue was collected on a sintered-glass funnel and washed with water, then with 0·1 per cent. hydrochloric acid, water, and alcohol. After a final treatment with ether, it was filtered and left in the air to dry. The yields of both samples were 77·2 per cent. of the dry weight of holocellulose taken.

The 5 per cent. sodium hydroxide fraction was prepared by first treating two 7·5 g. samples of holocellulose with 0·2 per cent. sodium hydroxide as described above. The residue was then washed with water, drained, and weighed, and aqueous sodium hydroxide was added to give 150 ml. of a 5 per cent. solution. After extraction for two hours, the material was collected on a sintered-glass funnel and washed with 20 ml. of 5 per cent. sodium hydroxide, then with water, 0·1 per cent. hydrochloric acid, water, alcohol, and ether. The yields were 66·4 per cent. and 66·1 per cent.

In order to obtain the 10 per cent. sodium hydroxide fraction, the procedure for the preparation of the 5 per cent. fraction was followed, except that aqueous sodium hydroxide was added to give a 10 per cent. solution instead of a 5 per cent. solution. The product was washed in turn with 20 ml. each of 10 per cent., 2·5 per cent., and 1·25 per cent. sodium hydroxide, and then with water, hydrochloric acid, alcohol, and ether, as before. The yields were 56·3 and 56·6 per cent.

A fourth fraction was prepared by treating holocellulose with boiling water.

Molecular weight distribution curves were obtained for each of these four factions according to the method of Ekenstam, and they are shown in the accompanying figures.

3. General Considerations

Fig 1 expresses the results of the fractionation as curves in which the amount of material dissolved is plotted against the strength of acid used. Figs. 2 and 4 show the integral weight distribution curves. If $f(P)dP$ denotes the number of all molecules having a polymerization degree between P and $P + dP$, then $Pf(P)dP$ measures the total amount (by weight) of the material which is present in this degree of polymerization. Hence the curves in Figs. 2 and 4 represent $F(P) = \int_0^P Pf(P)dP$. In order to get the weight distribution function $g(P) = Pf(P)$, it is necessary to differentiate the integral weight distribution curves and plot the result against P. The curves so obtained are the differential weight distribution curves. Figs. 3 and 5 show curves which are the result of differentiating the integral weight distribution curves of Figs. 2 and 4 and plotting the results against P.

It can be seen from Fig. 1 that about 74 per cent. of the holocellulose did not dissolve in the strongest acid used, i.e., 74 per cent. consisted of chains with a degree of polymerization greater than 1,200. Ekenstam's method only gives information on the form of the distribution function at chain lengths up to 1,200, and does not allow the completion of the $g(P)$ curve. Hence the differential weight distribution curve for holocellulose shows the distribution in holocellulose of those chains with a degree of polymerization less than 1,200. Similarly, the curve for the boiling-water fraction represents the distribution of chain length in only 40 per cent. of the water-extracted holocellulose.
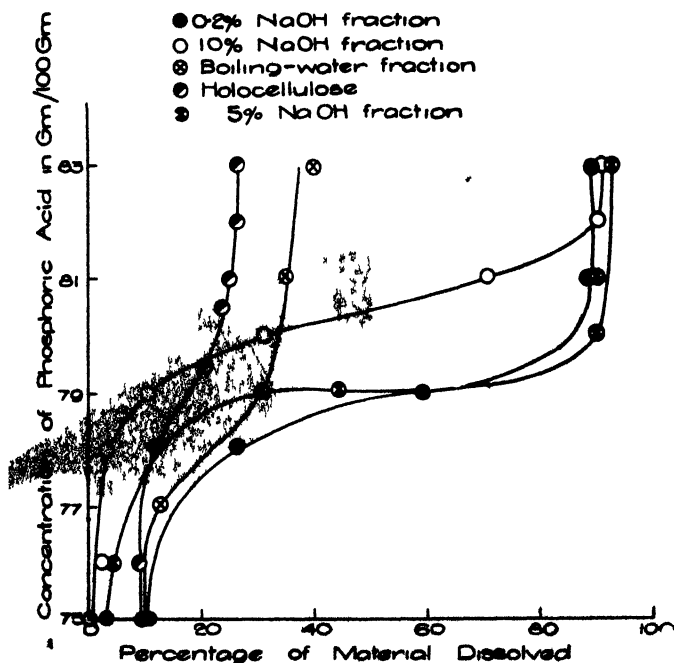
FIG. 1.—Fractionation curves.

By fractionation of spruce holocellulose, using the fractional solution method of Delmetsch and Reinecke, Atchison (1943) obtained a differential weight distribution curve containing two maxima and suggested that the carbohydrate existed in two " colonies." The average degree of polymerization for spruce holocellulose represented a very heterogeneous material which consisted of portions of very low and very high molecular weight, with only a small percentage having intermediate values. Distributions of the " two-colony " variety similar to that of Atchison have been reported (Coppick, 1944) also for rayons prepared from wood pulp as well as the original bleached sulphite pulp from which the rayons were prepared. Little, or no, " two-colony " effects were observed for bleached cotton linters, or for the rayons prepared from these.
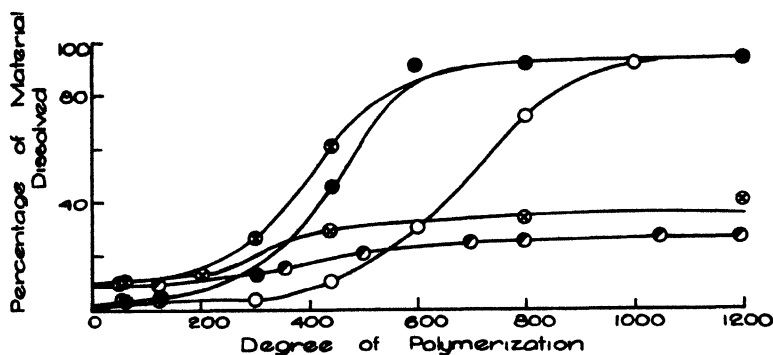
FIG 2.--Integral weight distribution curves *
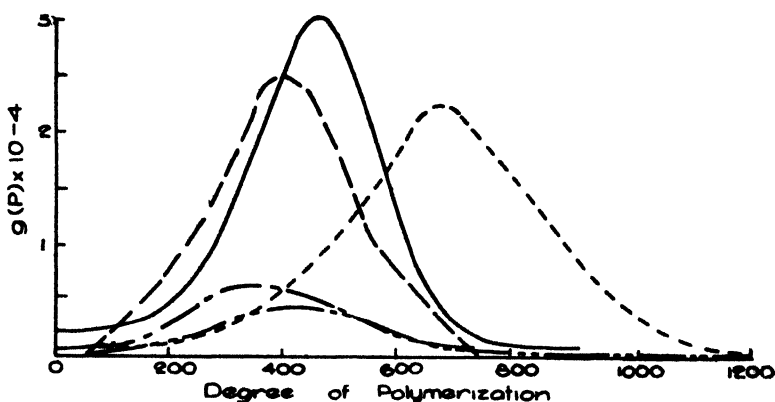
FIG. 3.—Differential weight distribution curves *

Since the holocellulose curve represented in Figs. 3 and 5 shows the results of fractionation for only about 26 per cent. of the holocellulose with a degree of polymerization below 1,200, it is possible that the curve forms the first hump of a " two-colony " curve similar to that of Atchison.

* For key, see graphs on next page

From Table 1, it can be seen that 15·7 per cent. of the holocellulose dissolved on treatment with boiling water. Of the residual 84·3 per cent., 40 per cent. had a degree of polymerization less than 1,200, and 60 per cent. had a degree of polymerization greater than 1,200, i.e., as a result of the treatment, 15·7 per cent. of the original holocellulose dissolved, while 33·7 per cent. was left with a degree of polymerization less than 1,200 and 50·6 per cent. with a degree of polymerization greater than 1,200. The curves in Figs. 3 and 5 show the distribution function for degress of polymerization up to 1,200, so that the curves for the boiling-water fraction only give the distribution in chain length for the material which represents 33·7 per cent. of the holocellulose. These results may be interpreted as showing that the d'fferential weight distribution curves for the boiling-water fraction, shown in Figs. 3 and 5, form the first humps of " two-colony " curves. Similarly, the curves for the three sodium
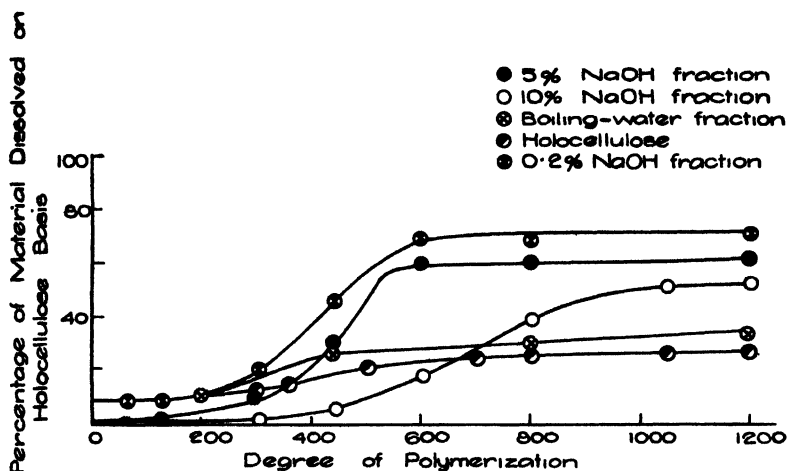
FIG. 4.—Integral weight distribution curves
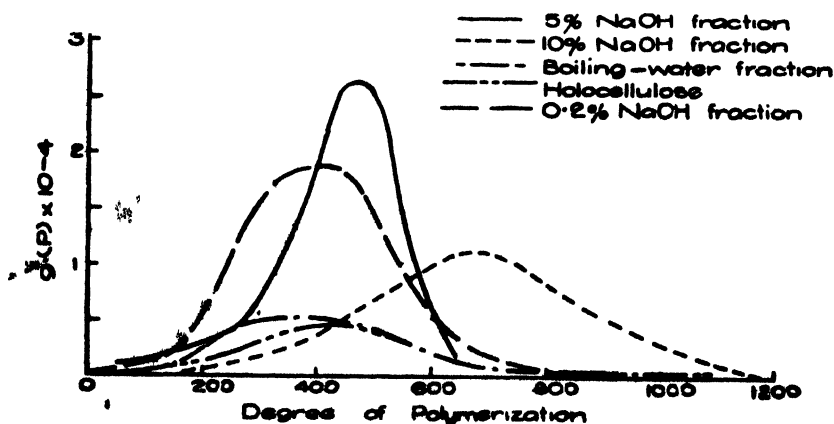
FIG. 5.—Differential weight distribution curves.

hydroxide fractions give the distribution in 69·5, 61·9, and 51·5 per cent. respectively, of the holocellulose, and form the first humps of "two-colony" curves.

TABLE 1.

Treatment of Holocellulose.	Residue (Percentage of Holocellulose)	Amount of Holocellulose Dissolved by Treatment	Percentage of Residue with D P* less than 1,200	Percentage (on Holocellulose Basis) of Residue with D P less than 1,200	Percentage of Residue with D P greater than 1,200	Percentage (on Holocellulose Basis) of Residue with D P greater than 1,200
Holocellulose . .	..	..	26·4	26·4	73·6	73·6
Boiling water	84·3	15·7	40·0	33·7	60·0	50·6
0·2 per cent. sodium hydroxide	77·2	22·8	90·0	69·5	10·0	7·7
5 per cent. sodium hydroxide	66·3	33·7	93·3	61·9	6·7	4·4
10 per cent. sodium hydroxide ..	56·4	43·6	91·3	51·5	8·7	4·9

* D P = Degree of polymerisation.

The graphs show that treatment with boiling water caused a slight increase in the size of the first holocellulose colony. The 0·2 per cent. sodium hydroxide extraction caused a very marked increase in the size of the first hump, the second hump being very much reduced so that it represents the distribution in only 7·7 per cent. of the original holocellulose. The 5 per cent. and 10 per cent. alkali treatments brought about further increases in the first hump, the second being correspondingly reduced and showing the distribution in only 4·4 and 4·9 per cent. respectively of the holocellulose. With an increase in the strength of alkali used in the extractions, the mean value for the degree of polymerization in the distributions represented by the first hump increased, while the second hump decreased, until, on treatment with 10 per cent. caustic soda, the second hump became very small, and the first assumed a form similar to that of bleached pulps. The fact that the mean degree of polymerization for the first colony increased as the strength of alkali increased indicates that, as the alkali treatment became stronger, the chains of the second colony were ruptured into fragments somewhat longer than the original members of the first colony of which they then became part.

Some consideration will be given now to the holocellulose determination, since this has some bearing on the discussion in the following section.

In the preparation of the *E. regnans* holocellulose used in the work described in this paper, the chlorinating medium was an aqueous solution of chlorine. Chlorine, in water, undergoes the reversible hydrolysis:

$$Cl_2 + H_2O \rightleftharpoons H^+ + Cl^- + HOCl.$$

Hypochlorous acid, formed by the hydrolysis, may dissociate according to the equation $HOCl \rightleftharpoons H^+ + OCl^-$. It is evident that the degree of acidity or alkalinity of an aqueous solution of chlorine greatly

affects its composition. The solution used in the holocellulose determination contained 6 g. per litre of active chlorine, and had a pH of 1·8. Under these conditions, there was a large amount of molecular chlorine and hypochlorous acid, but practically no hypochlorite ion. The hydrogen ion concentration of an aqueous chlorine solution also affects the intensity of its oxidizing activity, as measured by its oxidation potential. Of the three forms taken by chlorine in aqueous solution, the most effective from the standpoint of oxidation is hypochlorous acid, molecular chlorine being somewhat less active, and hypochlorite ion markedly less so.

Chlorine reacts with lignin in at least two distinct ways. The first of these, a chlorination reaction, takes place very rapidly, the velocity depending on the acidity of the solution. The second reaction is one of oxidation, and proceeds at a much slower rate, although it is more rapid in acid than in alkaline solutions. In treating lignified cellulose material with chlorine water, the hydrolysis of chlorine and the reaction with lignin take place very rapidly, accompanied by an increase in hydrogen ion concentration. Under these conditions, the lignin is halogenated to practical saturation within a few minutes, the oxidation reaction being so much slower that little oxidation can take place. The chlorine-lignin derivative is readily soluble in dilute alkali and may be removed by alkaline extraction. If the quantity of chlorine added to the material is greater than that which can be consumed by direct reaction with lignin, such excess is used up by oxidation of the organic matter present, including the chlorinated lignin products in solution, the chlorinated lignin remaining on the fibres, as well as the cellulose itself. Data of Voigtman (1933) show that use of excessive quantities of chlorine during the chlorination stage of sulphite pulp bleaching leads to a sharp drop in cuprammonium viscosity. The results of various workers (Wise, 1944, p. 744) indicate that acid hydrolysis of cellulose as a result of the acid conditions present during chlorination is not an important factor in cellulose degradation.

4. Discussion

The cellulose fabric in the secondary cell wall of plants forms a system which consists of a matrix of cellulose fibrils. This matrix is interpenetrated by a system of inter-connecting capillaries in which are deposited the amorphous cell wall constituents, mainly lignin and polysaccharides described as hemicelluloses. Similarly, each fibril consists of a complex network of micelles interspersed with a sub-microscopic system of canals containing amorphous material. There is a gradual transition from the crystalline to the amorphous regions, with the transition boundaries consisting of a mixture of cellulosic and hemicellulosic material.

Hemicelluloses are polysaccharides of at least two types, with different roles and from different locations in the cell wall. Under the general title of hemicelluloses are included those amorphous polysaccharides that form part of the system interpenetrating and encrusting the cellulose fabric, as well as the cellulosans that form part of the cellulose fabric itself.

One theory concerning the structure of wood celluloses envisages their intimate association as a special case of mixed crystallization in which the component molecules are chains consisting either of glucose units, as in cellulose, or xylose units, as in the cellulosans (Astbury *et al.*, 1935; Norman, 1937, p. 23). These chains are orientated and distributed at random through the micelle, the main longitudinal stability of which is provided by the long-chain cellulose components. According to Norman (1937, p. 28), there may be mixed chains of xylan and cellulose. It is not suggested that the units are jumbled along the chains, but that imperfect chains might occur, the whole length of which would be indistinguishable from that of a true cellulose chain. Schmidt's theory demands a chemical linkage and regular quantitative relationship between true cellulose and cellulosan (Schmidt, 1932). As a result of investigation of red beech, he held that there was a definite and constant relationship between the amounts of " difficultly-soluble " xylan and cellulose, these two being combined in an ester linkage. He was of the opinion that there were two xylan molecules linked with every cellulose molecule. No other workers have found such a simple stoichiometric relationship, and Lüdtke, specifically investigating the ester theory, was unable to find evidence supporting it.

The action of alkali solutions on pure cellulose will now be considered. The closely-packed, crystalline arrangement of the long cellulose molecules in the fibre makes it difficult for reacting molecules to enter between them, and necessitates an expansion of the crystal lattice and a breaking of the bonding between various hydroxyl groups on one chain to hydroxyl groups or ring oxygens on adjacent chains by hydrogen bridges. Reactions with cellulose are therefore of a highly heterogeneous nature, this being due to the long-chain molecular structure, and the micellar and fibre structure, which leads respectively to a random arrangement of substituted groups along the chains of glucose residues, higher substitution in the outer chains of the crystallites, and higher substitution in the outer sections of the fibres. Wise, in discussing the mechanism of cellulose-derivative formation, states, " The changes in X-ray diffraction patterns during derivative formation are generally explained on the basis of a micellar heterogeneous reaction. The reagent is assumed first to penetrate the fibre through its intermicellar spaces, and then to attack the cellulose chains on the surfaces, proceeding inwards, so that there is a progressive change from a completely reacted surface, through a partially reacted area to an unreacted interior."

There is evidence to support the fact that some micelles may react to a greater extent than others, and, furthermore, microscopic examination of cellulose fibres at different stages of reaction shows that reaction starts on the surface of the fibre and proceeds inwards in progressive stages. .

When a cellulose fibre comes into contact with water or other liquids, although adsorption may be regarded as the first process which occurs, the phenomenon of swelling is closely related to it. A distinct change of the X-ray diagram of the fibre is believed to indicate that the liquid has advanced into the interior of the chain bundles, so producing intramicellar swelling, and entered into a

reaction which results in the formation of a compound between cellulose and the swelling agent. The lack of a distinct change in the X-ray pattern generally indicates that the molecules of the liquid have attached themselves only to the outer surface of the fibre and to the interfaces between the micelles, producing intermicellar swelling. In the case of water, X-ray analysis of the cellulose-water system is interpreted to show that penetration is only as far as the surfaces of the micelles (Heuser, 1944, p. 42). The penetration is accompanied by swelling, which sets in as soon as the water enters the intermicellar spaces. Further penetration into the intramicellar spaces occurs only if the crystalline structure is essentially loosened and widened, as in the case when the fibre is treated with a strong swelling agent.

Whereas water causes intermicellar swelling in cellulose, this effect is enhanced with alkali solutions. Below a certain alkali concentration, the penetration into the spaces between the chains is regarded as very limited (Wise, 1944, p. 196; Heuser, 1944, p. 67). Thus, below concentrations of about 10 per cent., it is considered that only intermicellar swelling occurs, and that the alkali penetrates the amorphous regions, being taken up as well by adsorption or reaction on the surfaces of the micelles. With increased alkali concentration, intramicellar swelling occurs with the formation of alkali cellulose, showing an expanded crystal lattice in which there is a rupture of the bridging hydrogen bonds. This view is supported both by X-ray analysis (Katz and Seiberlich, 1940; Heuser, 1944, p. 68) and chemical methods (Heuser, 1944, p. 69).

Consideration of Table 1 shows that whereas more than 70 per cent. of holocellulose and 60 per cent. of the residue left after treatment with boiling water consisted of chains of a degree of polymerization greater than 1,200, not more than 10 per cent. of the three residues left after treatment with 0·2, 5, and 10 per cent. sodium hydroxide solutions respectively was made up of chains longer than this. It can be seen, therefore, that, whereas boiling water had little effect in reducing the large number of long cellulose chains in holocellulose, alkali as weak as 0·2 per cent. reduced the amount of long-chained holocellulose by about 60 per cent., and the alkali solutions of higher concentration had practically the same effect.

The boiling-water treatment removed 15·7 per cent. of the holocellulose, while the 0·2 per cent. alkali solution removed 22·8 per cent. Hence, the 0·2 per cent solution removed an extra 7·1 per cent. of the original material, at the same time causing a very considerable decrease in the chain length. This decrease must be due to one of two causes. Either the actual treatment with weak alkali brought about, in some way, the rupture of some of the linkages in the cellulose chains; or the removal of part, or all, of that material included in the 7·1 per cent. caused breakdown of the chains. The latter case will be considered first.

The material removed by boiling-water and dilute-alkali treatment consisted of polyuronides and xylan. If the removal of polyuronides caused the breakdown of the chains, the polyuronides would have to be associated with the cellulose in one of two ways suggested at the beginning of the discussion, i.e., they would necessarily either form part of the cellulose chains, or be included in the micelle as separate

chains. The presence of polyuronides in the micelle is not likely. According to Ott, the introduction of even a single polyuronide unit into the unit cell of the micelle would crowd it to the point of distortion (Ott, 1942). Xylan, then, remains as the only material whose removal may cause the decrease in chain length.

TABLE 2.

Treatment	Residue (Percentage of Holocellulose)	Xylan in Residue (Percentage of Holocellulose)	Xylan Removed (Percentage of Holocellulose)
Boiling water	84·3	13·9	8·0
0·2 per cent. alkali solution ..	77·2	11·9	10·0
5 per cent. alkali solution .	66·3	4·1	17·8
10 per cent. alkali solution .	56·4	1·2	20·7

Table 2 shows that the boiling water residue contained 13·9 per cent. of furfural-yielding material (calculated as xylan on a holocellulose basis), while the 0·2 per cent. alkali residue contained 11·9 per cent. The furfural-yielding content of holocellulose is 21·9 per cent., so that 8 per cent. of the original furfural-yielding material in holocellulose was removed by hot water, while 10 per cent. was removed by 0·2 per cent. sodium hydroxide. Thus if the decrease in chain length can be attributed to the removal of material by 0·2 per cent. alkali, the material must be all, or part of, the xylan in this extra 2 per cent. removed by the sodium hydroxide solution. Also, this xylan must be intimately associated with the cellulose, either as part of the cellulose chains, or as separate chains in the micelle.

It has already been pointed out that 0·2 per cent. alkali only penetrates the intermicellar spaces and amorphous regions of cellulose. If wood cellulose is considered to have xylan chains distributed throughout the micelle, this strength of alkali can only attack the xylan chains at the surfaces of the micelles or in the amorphous regions. The consequence of such an attack would be the freeing of accessible xylan chains, and their dissolution in amounts depending on their chain length. The removal of xylan in this way from such a cellulose-fabric model would cause no decrease in the amount of long cellulose chains.

If, however, wood cellulose is considered as having xylan units incorporated within the chains, a removal of xylan would be able to cause a marked decrease in cellulose chain length. As in the first case, the alkali on penetrating the intermicellar spaces and amorphous regions would be able to remove xylan units from these regions. Passing through the amorphous areas are many of the long cellulose chains which continue their length through these regions and several micelles. These chains will be in a position for attack by any liquid which penetrates the amorphous areas. If xylan units are distributed at random throughout the cellulose chains, many will be placed in those parts of the chain which pass through the intermicellar regions. An attack on the xylan in these positions and its consequent removal, would cause a decrease in the length of the chains from which it had

been removed. Thus, if the results revealed by the distribution curves are due to the removal of xylan by the 0·2 per cent. alkali solution, this xylan must form part of the chains themselves.

It is hard to see how the bonds at the junction of the xylan portion of the chain and the cellulose portion could be broken by the alkali treatment, as they are primary valence linkages, and one would expect that it would be no easier to break the linkage between xylose unit and glucose unit, than between two cellulose units.

The decrease in chain length produced by the action of dilute alkali may be due, not to the removal of any group from the cellulose chain, but to weak linkages in the chain itself. According to Horio* (private communication), who discussed the matter under consideration, " It is rather more natural to assume that there exist linkages in cellulose chains of holocellulose which are comparatively stable in acid, but particularly weak in alkali, and even in extremely dilute caustic alkali solutions."

The work of Davidson (1938) makes available experimental evidence for the discussion. He examined the relation between nitrocellulose fluidities and cellulose fluidities of oxycelluloses prepared from cotton cellulose by various treatments. He found that the oxycelluloses could be divided into two classes. The relation between the cellulose and nitrocellulose fluidities of those prepared by means of alkaline oxidizing solutions was almost the same as that between the two fluidities of hydrocellulose. It was not affected by alkaline treatment of the original oxycellulose. Those prepared with neutral or acid oxidizing solutions gave lower nitrocellulose fluidities than hydrocelluloses of equal cellulose fluidity, and the nitrocellulose fluidity of these might be increased even by cold treatments of the original oxycellulose with feebly alkaline solutions.

After boiling with dilute sodium hydroxide solution, all types of modified cotton gave the same relation between cellulose fluidity and nitrocellulose fluidity.

As an explanation of this observation, it was suggested that, in general, the oxidation of cellulose does not lead directly to the rupture of glycosidic linkages, but merely renders them extremely susceptible to hydrolysis by alkali. ' In the case of oxycelluloses formed in acid or neutral media, the original chain length is largely retained. Subsequent treatment of these oxycelluloses by alkali, however, causes a decrease in chain length, due to the breaking of alkali-sensitive glycosidic linkages. For oxycelluloses prepared by alkaline oxidants, this is not the case. The oxidation first leads to the production of linkages which are extremely sensitive to alkali, and these are then broken by the alkali present in the oxidizing mixture. If these oxycelluloses are given a subsequent treatment with alkali, their chain length is hardly affected.

Since the weakened glycosidic linkages are so easily broken by the action of bases, it is to be expected that they will be affected when oxycellulose prepared in an acid medium is dissolved in cuprammonium solution. In such cases the cuprammonium viscosity does not express

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the chain length of the oxycellulose as it results from the oxidation, but a chain length which has subsequently been shortened under the influence of the cuprammonium base.

In view of the suggestion given by Jackson and Hudson (1936, 1937), Davidson postulated that the mechanism of the shortening in chain length of oxycellulose produced by neutral or acid oxidizing agents involved the formation of a primary oxidation product in this manner:—

$$
O \begin{cases} \text{——CHO–Gl} \\ \quad | \\ \quad \text{CHOH} \\ \quad | \\ \quad \text{CHOH} \\ \quad | \\ \quad \text{CHO–Gl} \\ \quad | \\ \text{——CH} \\ \quad | \\ \quad \text{CH}_2\text{OH} \end{cases} \longrightarrow O \begin{cases} \text{——CHO–Gl} \\ \quad | \\ \quad \text{CHO} \\ \quad | \\ \quad \text{CHO} \\ \quad | \\ \quad \text{CHO–Gl} \\ \quad | \\ \text{——CH} \\ \quad | \\ \quad \text{CH}_2\text{OH} \end{cases}
$$
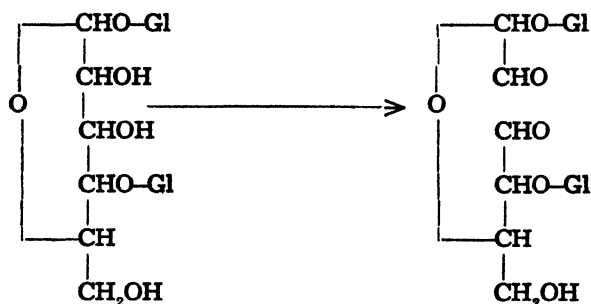
It was further assumed that the linkage between erythrose and the glucose radical of this primary oxidation product is susceptible towards the hydrolytic action of alkalis.

In the preceding section, it was mentioned that the chlorine water used in the preparation of holocellulose had a pH of $1 \cdot 8$, and under this condition contained large amounts of molecular chlorine and hypochlorous acid. As pointed out above, during the treatment of wood with chlorine, the lignin is very rapidly attacked and so exerts a protecting influence against the degradation of cellulose. If there is a large amount of chlorine water present, the excess is used up by oxidation of organic matter, including the cellulose, and so acts as an acid oxidizing agent upon cellulose. As such, the chlorine water is in a position to produce alkali-sensitive linkages in holocellulose. A solution of ammoniacal alcohol was used to dissolve the chlorinated lignin. This solution would break the alkali-sensitive linkages to some extent, but, according to Davidson (1938), the breaking action of ammonia is very much weaker than that of caustic soda. During the eight chlorination cycles used in the preparation of holocellulose, it is possible that alkali-sensitive linkages are accumulated, so the holocellulose might be regarded as an acid-oxidized oxycellulose. When this material is brought into contact with alkali, the solution would be able to break the sensitive linkages in the amorphous areas and on the surfaces of the micelles. This would lead to a shortening of the chain length.

5. Acknowledgments

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The Preparation of a Wood Sample for Chemical Analysis

*By A. J. Watson, A.M.T.C.**

Summary.

Investigations have been carried out on six different eucalypt woods to ascertain whether differences existed between 60-80 mesh and 60 — mesh samples for each species and whether there was any method of correlating values obtained from samples of different particle size.

It was established that differences did exist between the 60-80 and the 60 — mesh samples, such differences being consistent with losses of extraneous material from the 60-80 sample. In addition to these losses the greater ease of extraction of the finely ground material in the 60 — sample gives rise to marked differences in the values obtained for the 60-80 and the 60 — samples for determinations such as hot water solubility and alkali solubility. As no constant ratio existed between the values obtained for the 60-80 and 60 — mesh samples for those determinations which were considered to be independent of particle size, it was not possible to establish any relationship between these two fractions.

In the light of the data obtained it would appear that the 60 — mesh sample is satisfactory for the analysis of eucalypts provided that due attention is paid to the method of preparing the sample.

1. Introduction

The method employed in the preparation of a sample for analysis is of fundamental importance if the results are to be representative of the material being examined. This applies particularly to a substance such as wood, which has to be reduced to a finely divided state prior to analysis. A considerable amount of attention has been paid to this problem, both within the Division of Forest Products and in various overseas institutions. The problem of the preparation of samples for analysis from Australian woods has been discussed by Cohen and Mackney (1) who made an examination of samples prepared using either a mill with a cutting action (Wiley mill), or one with an impact action (Christy and Norris mill). Representative samples of two species were used for their investigation: *Eucalyptus regnans* F. v. M. selected as being representative of those woods which are practically free from extraneous materials, and *Eucalyptus marginata* Sm. as representing woods which contain large amounts of extractives. A number of samples of different particle size were prepared from both woods and then analysed for xylan content, lignin, and Cross and Bevan cellulose. As a result of their investigations the following conclusions were reached: —

    (i) Very fine comminution of the sample leads to some degradation of the Cross and Bevan cellulose during its isolation.

    (ii) It is possible to accept for analysis either the 60-80 mesh fraction or the 60 —† sample of *E. regnans*. The former is preferable because of uniform particle size, but in case

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† Throughout this paper the term 60 — sample is used to denote a sample passing a 60 mesh screen, 60-80 sample is the material that passes a 60 mesh screen but is retained on an 80 mesh screen.

of doubt where woods may contain larger amounts of extraneous materials, it would be safer to use the 60 — sample.

(iii) With samples of *E. marginata* it is not possible to disregard the fines in preparing a sample for analysis. Therefore the 60 — sample should be used.

As a result of these conclusions it was considered that it might be possible to divide the various woods into two groups according to the amount of extraneous materials that they contained. The 60-80 fraction would be used for the analysis of species low in extractives and the 60-sample for all those containing large amounts of extraneous materials. However, a series of analyses carried out on samples of marri (*Eucalyptus calophylla* R.Br.) showed that, even with the same species, samples could be obtained which differed widely in the amounts of extraneous materials they contained. It was obvious therefore that any question of dividing woods into groups, according to their extractives, was impracticable.

Apart from any question of the Cross and Bevan cellulose being partially degraded because of over-chlorination of the finely ground material in the 60 — sample, the fines in the sample made it more difficult to filter, with the result that the total time required for the determination was considerably prolonged. The need for avoiding over-chlorination of the finely ground materials has become more pronounced since 1939, when the paper by Cohen and Mackney was published, because an additional analytical procedure involving repeated chlorination treatments, viz., the holocellulose determination, has become a general routine practice for the analysis of woods in this laboratory.

One method which offered a possible solution to this problem was to use the 60-80 sample for all determinations involving chlorination treatments and the 60 — sample for all other determinations. It was realized that, particularly in the case of woods which contained large amounts of extractives, the composition of the 60-80 sample might differ from that of the 60 — sample, but it was considered that it might be possible to compare the 60-80 and 60 — samples on the basis of analytical data and, by establishing a ratio between the two samples, to calculate all results so that they could be expressed as a percentage of the 60 — sample. In order to ascertain the feasibility or otherwise of such a procedure, an investigation was planned using a number of different woods, ranging from those practically free from extraneous materials to those rich in extractives.

2. Experimental

The following eucalypts were selected for this investigation:— *E. regnans* F. v. M., *E. obliqua* L'Herit., *E. diversicolor* F. v. M., *E. marginata* Sm., *E. robusta* Sm., and *E. polyanthemos* Schau.

One specimen of each of these woods was selected and reduced to small chips. After these chips had been thoroughly mixed they were divided into two sections, both of which were milled in the Wiley mill until all the material, with the exception of 2 to 3 per cent., had passed the 60 mesh screen. The small amount of material that was rejected consisted almost entirely of particles which lodged

behind knives and on ledges in the Wiley mill, and as a consequence were retained in the mill. There was no reason to assume that this material differed in composition from the remainder of the sample. The 60, 80, and 100 mesh screens which were used in this experiment were tested for aperture size by the Munitions Supply Laboratories at Maribyrnong. It was found that the 80 and 100 mesh screens both conformed to international standard specifications, but that the aperture of the 60 mesh screen was approximately 11 per cent. larger than that specified, i.e., 0·0111 inches instead of 0·0099 ±0·0005 inches. The milling of the sample was carried out by feeding the chipped wood into the mill with the 1 mm. screen in position. All subsequent milling of the oversize material retained on the 60 mesh screen was performed using the 0·75 mm. mill screen. The oversize material was returned to the mill until only 2 to 3 per cent. remained on the 60 mesh screen and in the mill.

The following determinations were carried out on both the 60-80 and the 60 — mesh samples: percentage soluble in hot water, percentage soluble in 0·1 normal alkali, alcohol solubility, lignin (2), furfural content (3), methoxyl content (4), ash content and alkalinity of the ash (5), while Cross and Bevan cellulose (6), holocellulose (7), and furfural in holocellulose (3) were determined only on the 60-80 sample. The above determinations were carried out in duplicate, with the exception of the furfural yield, which was determined in triplicate, and the Cross and Bevan cellulose and holocellulose, for which the determinations were carried out in quadruplicate.

The values obtained for the various determinations on both the 60-80 and the 60 — samples for the six woods are set out in Table 1.

3. Discussion

It was assumed in this investigation that those determinations in which the structure of the wood was destroyed during the course of the determination were independent of the particle size. In these determinations any differences in the values obtained for the 60-80 and the 60 — samples should be due only to variations in their composition. Consequently the ratio of the 60-80 fraction to the total sample (60 —) should be a measure of any differences in composition between these two samples. It was considered that the following determinations would not be influenced by particle size: furfural yield, methoxyl content, ash content, alkalinity of ash, and lignin content. Of these determinations it was found that only the first two gave values which permitted any definite conclusions to be drawn. The values obtained for the ash content and the alkalinity of the ash were so small that the experimental errors involved prevented any definite conclusions being drawn from these results. Although the lignin determination is independent of particle size, at least within the limits of the present experiment, the actual value may have been influenced by the alkaline pretreatment which is definitely influenced by particle size. Even in the case of the furfural yield there is evidence (unpublished work) that the particle size may influence the total furfural evolved. However, this difference was detected, not between wood samples which closely resembled each other in particle size, such as the 60-80 and 60 — samples used in the present experiments, but between materials

TABLE 1.—SHOWING ANALYTICAL DATA OBTAINED ON 60-80 AND 60 — SAMPLES FOR VARIOUS WOODS.

(Only columns 6, 7, and 13 have been subjected to statistical analysis.)

Species	Sample Size	Ash	Alk. of Ash*	Solubility in—			Pentosan as Furf	Lignin	C. & B. Cellulose	Holocellulose	Furfural in Holocellulose		Total Methoxyl
				Hot Water	N/10 Sod Hydr	Alcohol					Percentage of Holocellulose	Percentage of Wood	
(1)		(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
		%		%	%	%	%	%	%	%			%
E. diversicolor	60—	0·024	0·69	7·8	21·0	7·6†	11·18	17·1	..	..	..	..	6·12†
	60-80	0·020	0·54	6·1	19·4	6·7	11·11	17·3	51·0	76·0	14·55	11·05	6·39
E. marginata	60—	0·040	0·02	7·1	28·8	4·5†	6·98‡	21·9	..	..	..	..	6·56‡
	60-80	0·013	0·02	5·6	27·0	3·6	7·11	22·4	46·2	68·5	10·76	7·37	6·80
E. obliqua..	60—	0·020	0·02	10·6	19·8	8·5†	7·82	19·1	..	..	..	..	6·79
	60-80	0·012	0·01	7·7	16·5	6·8	7·95	20·6	56·2	75·1	9·99	7·56	7·12
E. polyanthemos	60—	0·040	0·05	17·7	32·2	17·3†	7·27†	21·2	..	..	..	..	6·02
	60-80	0·035	0·06	14·5	28·8	14·8	7·54	21·7	40·3	66·5	10·77	7·15	6·79
E. regnans	60—	0·055	0·11	4·4	15·4	3·4	10·71	20·0	..	..	..	..	7·16
	60-80	0·045	0·10	3·5	14·6	3·2	10·79	19·4	59·5	73·6	13·54	10·00	7·28
E. robusta..	60—	0·131	0·22	14·5	29·1	15·1†	7·63	23·1	..	..	..	..	6·79
	60-80	0·098	0·25	12·4	25·2	13·0	7·77	22·8	45·5	65·3	11·42	7·48	6·92

* Expressed as ml. of N/10 acid per gram of O.D. wood.

† Differences between 60-80 and 60 — samples significant at the 1 per cent. level.

‡ Differences between 60-80 and 60 — samples significant at the 5 per cent. level.

widely different in particle size, such as wood chips and finely ground
wood. Consequently any difference in the furfural yield from the
60-80 and the 60 — samples, due to variation in particle size, could
be expected to be extremely small.

An examination of the values obtained as set out in Table 1 makes
it evident that there were differences between the 60-80 mesh and
the 60 — mesh material for all the samples examined. With two or
three minor exceptions all differences between these two samples were
consistent with the 60-80 sample containing less extraneous material
than the total sample, as represented by the 60 — material. These
differences can be seen more clearly if the furfural yields and the
methoxyl contents of the 60-80 and the 60 — samples are examined.
A statistical analysis of these results indicated that the values
obtained for the 60 — samples were on the whole significantly lower
than those on the 60-80 samples. These differences, however, were
quite small and in some cases were not significant when the results
from individual species were compared. Also the ratio of the values
obtained for the 60-80 samples to those of the 60 — samples was not
the same for the furfural yield as for the methoxyl content.

The determinations in which particle size might be expected to
exert some influence, viz., solubility in sodium hydroxide or hot water,
showed much greater differences between the 60-80 and the 60 —
samples. This is not surprising because they were both determined
under empirical conditions, namely, extraction in a boiling water bath
for one hour. It was only to be expected, therefore, that the hot water
or alkali soluble materials would be removed more rapidly from the
smaller sized particles, thus giving a higher extraction value for the
60 — sample. This point was checked by taking some of the 60-80
mesh material for each wood and milling it in the Wiley mill until it
passed an 80 mesh screen. This was screened so as to give 80-100 and
100 — mesh fractions. The 60 — sample for all the woods was
screened and the relative amounts of 60-80, 80-100, and 100 — mesh
material noted. The various fractions from the ground 60-80 sample
were then mixed in the same proportions. In this way a sample
which resembled the 60 — material in particle size was prepared from
the 60-80 sample. As no particular fraction had been rejected, any
differences between the samples could be due only to particle size.
The values for the hot water solubles in the original 60-80 sample
and the same material after milling to resemble a 60 — sample, as
far as particle size was concerned, are shown in Table 2. Statistical
examination indicated that, with the exception of *E. marginata*,
significant differences existed between the values obtained for these
two samples. These results demonstrated quite definitely that two
factors were involved in the different values obtained, (*a*) the loss of
finely ground materials, which are rich in extractives, from the
60-80 sample, and (*b*) the greater ease of extraction from the finely
ground material in the 60 — sample.

The determination of solubility in alcohol is carried out in a
Soxhlet extractor until no further colouring material is removed. Any
differences in alcohol solubility between the 60-80 and the 60 —
samples should therefore be due only to differences in their composi-
tion. In this case the ratios of the material insoluble in alcohol rather

than the alcohol solubility must be considered. These ratios, together with those obtained from the furfural, and methoxyl determinations, are shown in Table 3.

TABLE 2.—SHOWING PERCENTAGE SOLUBLE IN HOT WATER IN THE 60-80 SAMPLE AND IN A PORTION OF THE SAME SAMPLE AFTER MILLING TO GIVE A SIMILAR RANGE OF PARTICLE SIZE TO A 60 — SAMPLE.

Species *				Percentage Soluble in Hot Water.	
				On 60–80 Sample.	On 60–80 Sample Milled to Resemble 60– Sample.
E. diversicolor	..	..	..	6·1	6·5‡
E. marginata	..	..	..	5·6	5·9
E. obliqua ..	..	..	.	7·7	8·6†
E. polyanthemos	..	..	..	14·5	16·2†
E. regnans ..	..	..	..	3·45	4·40†

* Not sufficient 60-80 sample of *E. robusta* available for analysis.
† Differences significant at the 1 per cent. level.
‡ Differences significant at the 5 per cent. level.

TABLE 3.—SHOWING RATIOS OF FURFURAL YIELD, METHOXYL CONTENT AND MATERIAL INSOLUBLE IN ALCOHOL AS DETERMINED ON 60 — AND 60-80 SAMPLES.

Species.			Ratio $\left\{ \dfrac{60-}{60-80} \times 100 \right\}$		
			Furfural Yield.	Methoxyl Content.	Material Insoluble in Alcohol.
E. diversicolor	..	..	100·3	95·8	99·1
E. marginata	..	.	98·2	96·8	99·1
E. obliqua ..	..	..	98·4	95·4	98·2
E. polyanthemos	..	..	96·5	97·5	97·2
E. regnans ..	..	..	99·4	98.4	99 7
E. robusta ..	..	..	98·9	98.1	97·5

As these three determinations are the only ones which may enable results obtained on a 60-80 sample to be calculated to a total wood basis, as represented by the 60 — sample, it is of interest to examine these ratios. It is evident that there was little agreement between the ratios obtained from any of the three determinations. It is improbable that all these differences can be attributed to experimental errors, as the replicates in all three of these determinations agreed very closely. In general the ratios for the material insoluble in alcohol, and the furfural yield, follow a similar trend, but there is no similarity between the ratios of either of these determinations and that obtained from a study of the methoxyl contents of the 60-80 and the 60 — samples. It is difficult to find any explanation to account for the differences between these three ratios, and to decide whether any one ratio is more truly representative of the differences between samples than any other. The same type of variation between ratios

was observed when the ratios of the furfural yield and the apparent lignin content were compared for samples of different particle size from *E. regnans* and *E. marginata* (1). At the present stage, therefore, it is difficult to see any simple method for converting results obtained on a 60-80 sample to a 60 — basis, although it is evident that determinations done on the 60-80 sample are somewhat higher than they would be for a truly representative sample.

There is one further important point that arises from the present investigation. It has been assumed that the 60 — sample was the most suitable for the chemical analysis of wood, with the exception of the Cross and Bevan cellulose and the holocellulose determinations, because, as it represented the total sample, there was no danger of any portion of the wood sample being rejected. It has been demonstrated, however, that the particle size has a marked influence on the hot water solubility, and it is highly probable that the alkali solubility will also be influenced by this factor. As the lignin is usually determined on wood which has previously been extracted with alkali the value obtained will also be influenced by the particle size.

This factor does not appear to be particularly serious if a Wiley mill is used for the preparation of the wood samples. The following table shows the various fractions obtained on milling two matched samples of *E. regnans*.

TABLE 4.—SHOWING INFLUENCE OF THE CONDITION OF THE WILEY MILL ON THE PARTICLE SIZE.

Condition of Blades.	Percentage of Various Fractions Obtained on Milling				
	+60.	60–80.	80–100	100–.	Loss.
Sharp	2·8	52·9	22·8	20·2	1·3
Blunt	6·1*	51·0	18·2	22·7	2.0

* This amount of oversize material could not be reduced, even on repeated milling.

The first sample was milled immediately after the mill had been resharpened and set, while the second was prepared after the blades of the mill had lost their edge and become dulled. It is evident, even when the mill has been used for a considerable period, that the percentage of 100 — material in the whole sample is not greatly increased. It would appear therefore that if a Wiley mill is used for the preparation of wood samples for analysis, the percentages of 60-80, 80-100, and 100 — mesh samples are not likely to vary sufficiently to influence the values obtained for hot water solubility or alkali solubility.

When these results are considered in conjunction with the Cross and Bevan cellulose results obtained by Cohen and Mackney (1) on 60-80, 80-100, and 100 — samples of *E. regnans* and *E. marginata* it would appear that, apart from some increase in total time required for the determination because of the slower filtering rate of the more finely ground material, the 60 — sample prepared in the Wiley mill is

satisfactory for all determinations. Experience in this laboratory has shown that the determination of holocellulose is not complicated to any great extent if a 60 — sample is used for its determination instead of a 60-80 sample.

As the attempts to find a common ratio between the 60-80 and the 60 — samples, which would have permitted the 60-80 sample to be used for all determinations involving chlorination treatments, were unsuccessful, it would appear that it would be best to use the 60 — sample for all determinations. This procedure would only be satisfactory if the sample were milled under carefully standardized conditions, so as to obtain a sample of uniform particle size. If a screening analysis were included with the analytical data it would ensure that the sample being examined was not milled in such a way as to give rise to erroneous results for those determinations such as Cross and Bevan cellulose, holocellulose, hot water solubility and alkali solubility, which are influenced by the presence of very finely ground material.

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Electrolytic Polishing of Copper in Orthophosphoric Acid

By R. W. K. Honeycombe, M.Sc.,* and R. R. Hughan †

Summary.

The brightening of copper anodes in aqueous orthophosphoric acid electrolytes was investigated by measuring the anode potential with a calomel half cell. By plotting the current density for different anode potentials, a characteristic type of curve was obtained, the horizontal portion of which defined the range of conditions over which polishing occurred.

The variables investigated included temperature, concentration of the electrolyte, and the disposition of the electrodes. The theories of Jacquet and Elmore are discussed and are shown to be inadequate in explaining the experimental results.

An appendix discusses briefly the optimum conditions for the electrolytic polishing of copper specimens for metallographic examination.

1. Introduction

The phenomenon of electrolytic brightening or polishing was probably first observed on anodes in electroplating baths many years ago, but until recently no attempt was made to investigate or to utilize the effect. Jacquet (1) first made a serious study of the phenomenon, and in 1935 published a method by which copper could be given a mirror finish by making it the anode under certain conditions in an orthophosphoric acid electrolyte. Jacquet later extended the method to brasses, bronzes, and copper-base alloys generally. Furthermore, he developed a series of perchloric acid—acetic anhydride electrolytes in which he polished tin and lead (2). In 1939, Jacquet and Rocquet (3) described a method of electrolytically polishing steels.

Jacquet's earlier work was mainly directed towards obtaining good polished surfaces on small metal specimens for subsequent metallographic examination. In parallel with such investigations there have been many atempts to apply this principle to industrial processes. These have been very successful in a number of cases, the outstanding applications in industry at the present time being the electrolytic polishing of stainless steels and aluminium. It is clear that these electrolytic methods of surface preparation will eventually play an important role both in the industrial treatment of metals and in the metallurgical laboratory (6).

The surfaces produced by electrolytic polishing are free from deformation and the Beilby layers which are normally produced during mechanical polishing. There is no risk of obtaining false structures as a result of deformation or recrystallization of surface layers, and the surface is entirely scratch free. In this laboratory, electrolytic methods of polishing have been extensively used for metallographic work and it has been found that the results obtained are in many cases superior to those obtained by mechanical polishing. The metals aluminium, cadmium, copper, lead, magnesium, tin, zinc,

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and also brasses (single phase and duplex), nickel-silver alloys and steels have been satisfactorily prepared. The methods used for these metals have been previously summarized (4, 6).

Recent work has shown that this method of surface preparation may play an important part in the measurement and investigation of friction and wear. In addition to developing routine methods of polishing various metals and alloys, it was thought desirable to study more systematically the phenomenon of electrolytic polishing in an attempt to throw some light on the mechanism by which it occurs. This paper describes an investigation into the electropolishing of copper in aqueous orthophosphoric acid electrolytes with this end in view.

2. Previous Work—Mechanism of Electrolytic Polishing

In his work on copper, Jacquet (1) found that if the cell voltage was plotted against the current density (in a cell of orthophosphoric acid with copper electrodes), a curve similar to that shown in Fig. 1 was obtained. Polishing of the surface did not commence till point C was reached; in the range AC etching occurred. The range over which good electrolytic polishing occurred was defined by the flat portion DE of the curve. Above E the current rose sharply as the voltage was increased, and gas bubbles commenced to form at the anode. Polishing occurred in this range but the surface was pitted as a result of the adherance of small bubbles to the surface. Jacquet recommended a certain composition (500g./litre) of electrolyte, but no detailed investigation of the variables—composition of electrolyte, effect of temperature, &c.—has yet come to our notice.

To explain the polishing of copper in orthophosphoric acid, Jacquet postulated the formation of a film on the anode surface, consisting mainly of copper phosphate. He suggested that the thickness of this film would be smallest at the tops of the " hills " on the metal surfaces and thus the resistance of the film at these points would be lower. This would cause preferential solution of the tops of hills as a result of the higher current density existing there and would lead to a
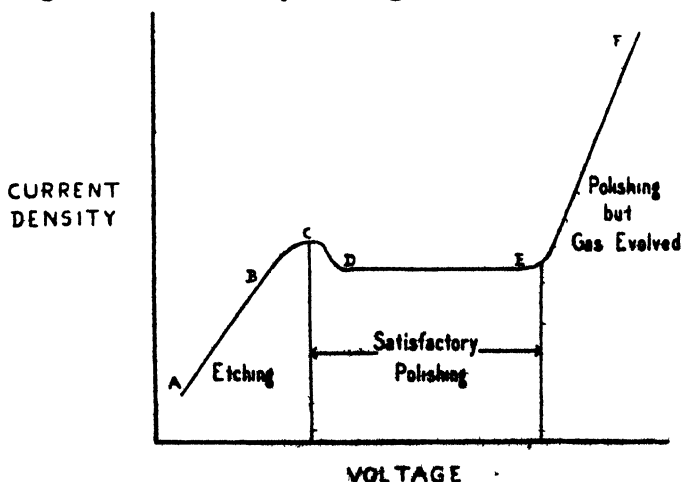
FIG. 1.—Typical current density-voltage curve obtained in an electrolytic polishing cell.

general smoothing of the surfaces. More recently, Elmore (5) has suggested that the rate of diffusion of ions through the film is the most important factor. This would of course be greater on the hill tops because of the greater concentration gradient existing there, and thus again preferential solution can be explained.

This paper describes the effects of varying the concentration and temperature of the electrolyte (orthophosphoric acid), and a technique of investigation in which the anode potential is measured, is described.

3. Experimental Method

The type of curve shown in Fig. 1 can only be satisfactorily obtained if a potentiometric type of circuit is employed. The resistance of the circuit should be kept low and small changes of resistance in the cell can then produce an appreciable change in current.

Most previous workers measured the voltage drop across the cell, but as the polishing process seems to be associated with a reaction at the anode it was thought that measurement of the anode potential might prove valuable. In this way the anode potential could be isolated from the cathode potential and the potential drop through the electrolyte.

In the experiments described below, the anode potential with respect to the solution was measured by means of a calomel half-cell, electrical contact with the electrolyte being made by means of a KCl-agar bridge. The anode potential was obtained by balancing the potential on a potentiometer. Fig. 2 shows a schematic arrangement of the polishing cell and circuit.
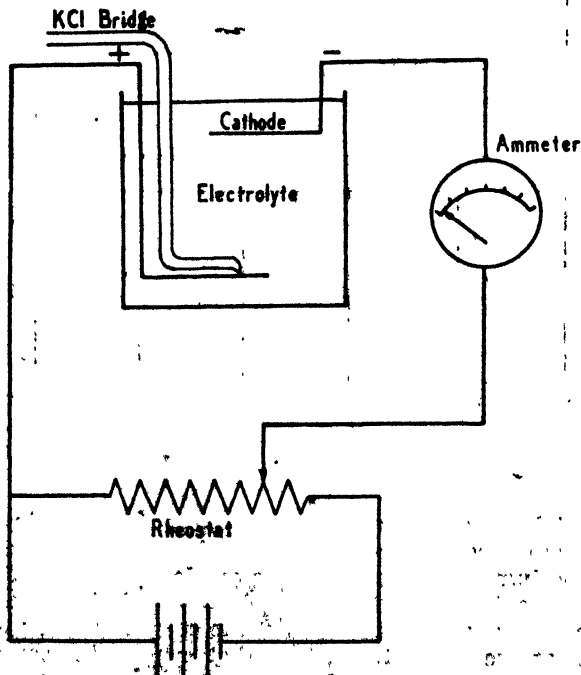
Fig. 2—Electrolytic cell and circuit used for experimental determination of curves.

Both the anode and cathode were made of copper sheet, the anode being underneath and $1\frac{1}{2}$ inches from the cathode. For most of the experiments the anode was coated with stopping-off lacquer except for 1 square inch on the surface facing the cathode. The area of the cathode was 10 square inches.

The effect of variables such as composition of electrolyte, temperature, distance between electrodes, and relative size of electrodes, was investigated by determining an anode potential—current density curve. This was done by increasing the voltage in small steps (0·1 volt) and allowing the cell to attain equilibrium before each reading of the current was taken.

4. Experimental Results

(1) *The Anode Potential-Current Density Curve.*

On plotting the anode potential-current density curve for a cell containing orthophosphoric acid of any concentration and with copper electrodes, the shape of curve obtained was similar to the voltage (across cell)—current density curves obtained by previous workers. Polishing occurred under conditions represented by the flat portion of the curve.

A comparison of the anode potential and voltmeter curves is shown in Fig. 3. As the size of the anode increases with respect to the cathode, the voltmeter curve deviates more and more from the

Volts.
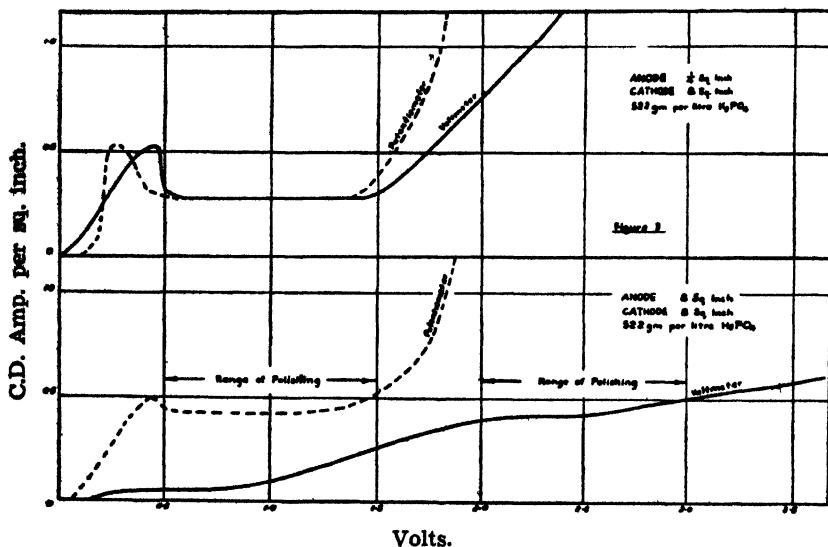
FIG. 3.

anode potential curve. The voltmeter curve becomes less ideal in shape and finally ceases to indicate a plateau. It should also be noted that the hump C (Fig. 1) in the anode potential and voltmeter curves is a convenient representation of the instability of current and voltage over this range. In actual fact the current-voltage or current-potential values are found to fluctuate between limits represented by a continuation of curves AB and ED.

(ii) *Effect of Temperature.*

Anode potential-current density curves were determined for an electrolyte containing 522 g./litre orthophosphoric acid at the temperatures of 4·5°, 22°, 31°, 50°, and 70°C. These curves are shown in Fig. 4. The curves were geometrically similar, the length of the
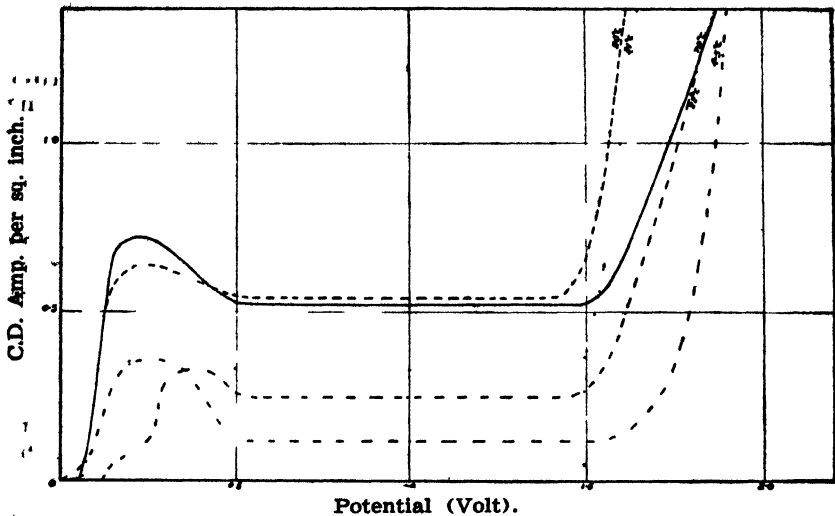
FIG. 4—Anode potential—current density curves at different temperatures   Electrolyte: 522 g./litre H_3PO_4

" plateau " being the same in each case, but occurring at a different current density. Copper specimens were polished for 10 minutes at different temperatures, and at a voltage representing the middle point of the plateau. The state of the surface was noted in each case (Table 1).

TABLE 1.—EFFECT OF TEMPERATURE ON POLISHING OF COPPER SPECIMENS.

Temperature	Anode Potential (Plateau)	Current Density	State of Surface
°C.	Volts.	Amps./sq. in.	
4·5	0·5–1·5	0·12	Good polish
22	0·5–1·5	0·25	,, ,,
31	0·5–1·5	0·33	,, ,,
50	0·5–1·5	0·54	Somewhat uneven
70	0·4–1·4	0·52	Uneven

(iii) *Concentration of Electrolyte.*

Aqueous solutions of orthophosphoric acid containing 65–1,570 g./litre were made up and anode potential-current density curves determined for each electrolyte using standard conditions—copper anode 1 sq. inch in area, copper cathode 10 sq. inches in area, placed 1½ inches above the anode. In all cases the voltage range of the

FIGURE 5.

Anode Potential — Current Density Curves

Temperature Approx. — 20°C

261 gm./litre.

196 gm./litre

65 gm./litre.

522 gm./litre.

1044 gm./litre.

1570 gm./litre
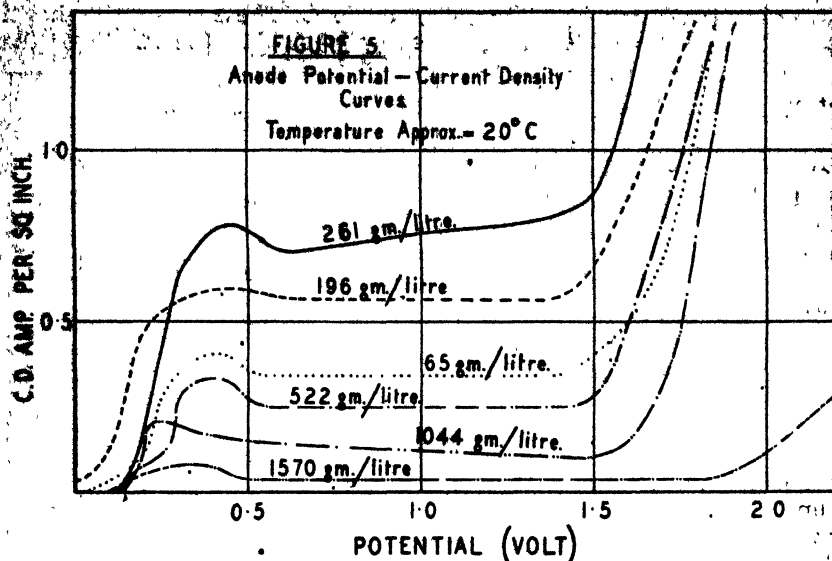
POTENTIAL (VOLT)

FIG. 5.—Anode potential—current density curves for different concentrations of electrolyte. Temperature 20°C. approx.

series of experiments, typical copper specimens were polished in each electrolyte using the voltage representing the middle of the " plateau." After ten minutes each specimen was removed and inspected. Table 2 summarizes the results obtained.

TABLE 2.—EFFECT OF CONCENTRATION OF ELECTROLYTE ON THE POLISHING OF COPPER.

Concentration g./litre H_3PO_4.	Anode Potential (Plateau).	Current Density.	State of Surface.
	Volts.	Amps./sq. in.	
65	0·3–1·2	0·34	Whole surface etched
130	0·4–1·5	0·64	,, ,, ,,
196	0·5–1·4	0·56	Signs of polish on edges of specimens
261	0·6–1·5	0·75 (av.)	Polishing on edges of specimen
392	0·5–1·5	0·33	Polished all over but poor at centre of specimen
522	0·5–1·5	0·25	Very good polish on entire surface
1044	0·3–1·5	0·125	Very good polish—original scratches not entirely removed
1570	0·3–1·8	0·04	Very good polish but original scratches not entirely removed

Etching of the surface of the specimens occurred in the more dilute solutions, and satisfactory polishing was not obtained until the concentration was raised to 500 g./litre orthophosphoric acid. The more concentrated solutions polished very well, but as a result of their lower conductance the process became much slower and a much longer time was required to remove the original scratches from the copper. The optimum concentration seemed to be in the vicinity of 500 g./litre.

(iv) *Distance Between Electrodes.*

Under the same conditions of temperature and concentration, the current density corresponding to the plateau did not vary as the distance between the electrodes was changed.

(v) *Disposition of the Electrodes.*

In the above experiments, the anode was placed horizontally beneath the cathode, because experience had shown that this gave the most satisfactory results. Polishing still occurs if the electrodes are arranged vertically but the current density at which polishing occurs is higher. Furthermore, streaks and furrows are often produced on the surface as a result of the streaming of a viscous liquid film from the surface of the anode downwards. Electro-polishing was also found to occur when the anode was rotated rapidly, but in these experiments no improvement in the state of the surface over that produced by stationary methods was observed, and a higher current density had to be employed.

5. Discussion

The experimental results show that the range of polishing of copper in orthophosphoric acid electrolytes can be defined by an anode potential-current density curve. This implies that the polishing occurs as a result of anode reactions and that reactions in other parts of the cell are not relevant. The evidence supporting the film theory first postulated by Jacquet and later modified by Elmore is as follows:—

1. The plateau in the anode potential curve indicates that there are limiting current conditions. These limiting conditions could be explained by the presence of a concentration gradient at the anode surface with a film saturated with copper ions or phosphate ions on the surface.

2. An increase in the temperature of the electrolyte raises the current density at which the plateau occurs. As the rate of diffusion of ions increases with temperature, it would be expected that a higher current density would be required to form a stable film at elevated temperatures.

3. Dilution of the electrolyte raises the current density of the plateau within certain limits. Again this can be explained by the increased diffusion of the ions from the film necessitating a higher current density to attain a stable condition.

4. A change in the distance between the two electrodes has no apparent effect on the critical current density. It thus appears that the resistance of the solution is small compared with the resistance in the vicinity of the anode surface, a state of affairs which is readily explained by the film theory.

The work of Elmore was based on two major assumptions. Firstly, that the metal ions leave the vicinity of the anode by diffusion and not by migration, since most of the current would be carried by the more mobile hydrogen ions. Secondly, he considered that the concentration layer at the anode surface is saturated and that the current is limited by diffusion of ions from this layer. These assumptions are in complete concordance with the formation of a plateau in the anode potential-current density curve. However, there are certain facts which are difficult to understand on this basis.

Firstly, the voltage range of the plateau does not alter with changing conditions of temperature and concentration (Figs. 4 and 5). It would be expected that a higher potential would be necessary to maintain an anode film as the conditions became less favourable for its retention at the anode surface. Furthermore, the presence of a plateau in the curve does not necessarily mean that polishing must occur. In very dilute solutions and in hot solutions the copper ceases to polish. Apparently in these cases, there is insufficient potential drop through the film to cause differential solution of the copper surface and thus produce polishing. However, Elmore's theory presupposes that the formation of a plateau in the curve indicates that the anode film is saturated. The'maximum occurring in the plateau current density—concentration curve is also difficult to explain.

It thus appears that the function of a saturated ionic layer is not the only factor contributing to the attainment of a limiting current. As oxygen is liberated when the maximum plateau voltage is exceeded it seems likely that an oxide layer exists at the anode surface. This layer may play a significant role in the production of a limiting current at the anode surface.

6. Acknowledgment

We wish to thank Mr. G. M. Willis for helpful discussions and criticism.

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Appendix

OPTIMUM CONDITIONS FOR ELECTROLYTIC POLISHING OF COPPER SPECIMENS.

In practice, it is often not convenient to determine anode potential-current density curves prior to electrolytic polishing of a specimen. If a potentiometric circuit is employed, the current density-cell voltage curves should indicate approximately the range over which polishing occurs. The beginning of the plateau is marked by an unstable region, and the end of the plateau is marked by the evolution of fine bubbles of oxygen from the anode. Metallographic inspection of polished specimens will also determine approximately the conditions under which they were prepared. Plate 2, Fig. 1, represents an area on a specimen prior to electrolytic polishing; the scratches are those produced by a 600 carborundum paper. Specimens treated below the minimum plateau voltage present an etched appearance (Plate 2, Fig. 2), while specimens polishing in the plateau range have a mirror-like surface which can be lightly etched to show up the grains (Plate 2, Fig. 3) by lowering the voltage to below the plateau for a short time. Specimens treated above the maximum plateau voltage are polished but the surface is pitted as a result of the presence of the fine gas bubbles (Plate 2, Fig. 4).

The optimum concentration of electrolyte for copper is about 500 g./litre orthophosphoric acid. For metallographic specimens not exceeding 3 square inches in area, a cell voltage of between one and two volts is necessary. Best results are obtained with the electrodes arranged horizontally and about 1—2 inches apart.

A Pneumatically Operated Caulking Gun

By B. M. Holmes, M.Sc.*

Summary.

The pressure caulking equipment described in this report was con-
structed with the object of increasing the efficiency of caulking operations
in the erection of precast concrete houses constructed by the Victorian
Housing Commission. The equipment has been tested under working
conditions on houses of this type with satisfactory results and should
find application wherever extensive use is made of caulking materials.

1. Introduction

The usual method of applying caulking compounds to buildings
in this country is by means of a knife or small tool. However, on
account of the intrinsic stickiness of these materials, caulking by
this means is a tedious and frequently messy operation and in recent
years overseas practice has tended towards the use of a simple hand-
operated extrusion gun to facilitate caulking operations. Since the
orifice through which the compound must be extruded is relatively
small and the mechanical advantage of the mechanism is low, it is
usually necessary to formulate a special " gun " grade mastic for
application by this means, the consistency of this grade being some-
what lower than that of " knifing " grades which are intended for
trowel application.

In this country such caulking guns have not been widely used and
local proprietary caulking compounds are generally formulated in a
" knifing " consistency. Several proprietary brands are in fact for-
mulated in even heavier consistencies than ordinary knifing grades,
the reduction in stickiness thus obtained rendering hand caulking
methods less arduous.

To obtain the advantages of gun application it therefore appeared
desirable to develop a device capable of handling knifing grade
materials, and the obvious solution was to employ air pressure to
achieve the greater force required to extrude materials of this con-
sistency. It should be stated that such a device is not novel and that
pressure caulking has on occasions been utilized by Messrs. Nonporite
Pty. Ltd., Victoria, and possibly also by other contractors. A new
type of filling device has been evolved, however, and in view of the
simplicity and economy of caulking by this method, it has been
thought worth while describing the equipment used in its entirety.

2. Apparatus

The caulking equipment comprises the following components†:—

(1) *Pressure Gun* (Plate 3).

The barrel of the gun consists of a 15-in. length of 10-gauge
seamless steel tube 1¾ in. inside diameter, threaded at either end
and fitted with a nozzle; a piston, comprising two leather cup washers

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* An officer of the Building Materials Research Section.
† Working drawings of these components are available and will be
supplied on application to the Officer-in-Charge, Building Materials
Research, C.S.I.R., Graham Road, Highett, Victoria.

mounted back to back at the end of a piston rod, and a pressure head. The pressure head mounts the high-pressure inlet valve, a relief cock, and is fitted also with a gland to take the piston rod.

The nozzle of the present gun has a rectangular orifice and is ground off with a 30° angle to facilitate even paying out of the mastic. This orifice has been found satisfactory for caulking joints of rectangular cross-section. However, circular and triangular orifices may be used with advantage in other situations; for example, in paying triangular fillets around window frames and the like.

(ii) *Pressure Filling Pot* (Plate 4).

The filler is a cylindrical vessel, 6½ in. internal diameter, capacity 1 gallon, capable of receiving air pressure. It is fitted with a tail piece (which may be attached by means of a union nut to the muzzle end of the gun barrel), a detachable cover plate (mounting the air inlet and the relief cock), and a conical spreader. The latter slides freely within the body of the filler, being held in an axial position by a stem working in a guide screwed into the cover plate. In use, the spreader is pressed into position on top of the charge in the pot and serves to prevent the applied air pressure from blowing through the mastic charge. Although the pressure required to discharge mastic from the filler into the gun is quite low, yet it is desirable, in view of the method of connection of the filler to pressure source, that the pot be tested to the maximum pressure to be used in operating the gun. In the present case the filler was tested at a pressure of 150 p.s.i.

The filling pot is essential to the economical operation of the pressure caulking gun. Hand-operated caulking guns are commonly loaded in the manner of a syringe by dipping the end of the barrel in the mastic and withdrawing the piston, but while this procedure is a feasible (though messy) operation with gun grade mastics, the greater stiffness of knife grade materials precludes satisfactory filling by this means.

It would be possible to load the gun by packaging the mastics in cartridge form encased in cellophane or other pliable wrapping, and this method of packaging has been adopted overseas to overcome gun loading difficulties. However, at present, Australian mastics are not available in this form and the filler here described offers a very satisfactory alternative to the utilization of specially packaged charges.

(iii) *A Source of Compressed Air or Suitable Inert Gas*.

In laboratory and field trials a 100-ft. cylinder of compressed air was used. A portable air compressor could be substituted, although the low cost of compressed industrial gases competes favourably with other sources of supply. (Compressed air 5s. per 100 cu. ft.; nitrogen 9s. 9d. per 100 cu. ft. Carbon dioxide at 4d. per lb. is cheaper than these but is troublesome to use on account of the cooling

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* Compressed oxygen should not be used on account of the risk of explosion attendant on contamination of high pressure connections with hydrocarbon materials.

effect accompanying expansion to lower pressures.) The flow of gas was regulated through a standard "Comweld" oxygen regulating valve. Dunlop ⅜-in. diameter 4-ply compressor hose was used for delivery of the gas to gun and filler.

(iv) *Hand Trolley.*

A small hand trolley was used to mount the several components listed above and to facilitate transportation of the kit from point to point during caulking operations (Plate 4, Fig. 1).

3. Operation

The filler is loaded with caulking compound by hand (Plate 4, Fig. 2).

The operation of loading the gun from the filler is performed in the following stages:—

(i) The nozzle of the gun is removed, the piston pressed full distance into the barrel, and the muzzle of gun coupled to the tail of the filler by means of the union nut.

(ii) With the gun relief valve open and the filler relief valve closed, compressed air is supplied to the filler by means of the cock in the filler supply line. Pressure is maintained until the piston is driven back to the limit of travel.

(iii) Air supply to filler is cut off and pressure released. Gun and filler are disconnected and mastic is struck off with a knife.

(iv) The nozzle is fitted to the gun and relief valve closed ready for operation.

As mentioned above, the pressure required in the filling pot for the purpose of loading the caulking gun is quite low, but it is convenient to operate this component from the same source as is used to supply the gun itself. Supply is drawn through a T-piece coupled to the reducing valve on the gas cylinder.

The operation of the caulking gun is illustrated in Plate 3, Fig. 2.

4. Performance

The rate of delivery of mastic by the caulking gun is, of course, dependent on the operating pressure, the dimensions of the orifice attached to the gun, and on the consistency of the caulking compound used.

In the field trials to which the gun has been subjected, it was found convenient to use a nozzle with a rectangular orifice 1 in. by 9/64 in. cross-section and 1 in. long (shorter dimension). It was found possible to extrude at satisfactory rates through this orifice all Australian proprietary caulking compounds, with the exception of the several heavy-bodied materials referred to in Section 1, using air pressures ranging from 90-150 p.s.i.

The consistencies of materials satisfactorily handled ranged from 140 to 270 (in terms of penetration units*, determined with an I.P.T. penetrometer operated with a 25-g. load for 5 seconds at 25° C.†). For purposes of comparison, the consistency of a sample of an English gun grade mastic tested was approximately 370, while the consistencies of the heavy-bodied Australian caulking compounds referred to above lay in the vicinity of 60 (figures expressed in penetration units as above).

Trials with the equipment described above have been made in the field on "V.H.C." type prefabricated concrete units erected on the Moorabbin and Ashburton Estates of the Victorian Housing Commission.

Some variation in the widths of wall joints was encountered when caulking these structures. Nominally these joints are ¼ in. wide and, as already stated, the nozzle of the present gun was designed for insertion in joints of this dimension, but occasionally it was found that the joint width was less than ¼ in. and insertion of the nozzle was not possible. Insertion of the nozzle to the full depth of the joint is desirable as more effective packing of the material against the boundaries of the joint is thereby achieved.

However, provided there was sufficient width to receive the extruded ribbon of mastic (9/64 in.), a satisfactory seal could be made by following the gun with a small tool to press the compound firmly into the joint. Narrower nozzles than the one described could, of course, be used, but it is considered that such a provision would be of little practical value since it has been found that caulked joints much below ¼ in. in width tend to give unsatisfactory service§.

A comparative test was made to determine the economy in time obtainable by caulking joints of the V.H.C. house with the caulking gun. Identical houses were caulked, (a) with the gun, and (b) by the hand-caulking method previously adopted by the Victorian Housing Commission‡. The houses selected were constructed with nine exterior

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* This method of determining the consistency of caulking compounds has not been found entirely satisfactory, considerable variation in the penetration index being noted over different points on the surface of a sample of material. Alternative methods of measuring the property have not as yet been explored and the figures quoted above are included merely to serve as a guide to the range of consistencies encountered.

† "Standard Methods for Testing Petroleum and Its Products." 6th Ed., pp. 255-7. Institute of Petroleum, No. I.P.-49/44. (London, 1945.)

§ Tregoning, J. J., et al.—Plastic calking compounds. U.S. Dept. Commerce, Building Materials and Structures Rep. B.M.S. 33, pp. 10, 14 (1940).

‡ In caulking these houses by hand one of the heavy bodied caulking compounds is used. This is rolled out by hand to a long "rope" which is then held against the joint and pressed home with a pointed tool. Some doubt is entertained regarding the efficiency of the bond obtained by this method: the fact that the material used may be worked by hand in this manner is indicative of the reduction that has been made in adhesiveness. However, this material was selected largely on account of its ease of manipulation, and although its deficiency in this respect was realized, the difficulty in handling softer materials was a practical bar to their use.

wall joints, seven of which ran the full height of the wall, 9 ft. 5¼ in., while the remaining two were corner window joints, the aggregate joint length approximating to one full wall joint of 9 feet. Caulking by each method was performed by one man unaided. Data relevant to this trial are presented in Table 1.

TABLE 1.

Item.	Gun Caulking	Hand Caulking.
1. Location of test	VHC Ashburton Estate Site No. 175	VHC Ashburton Estate Site No. 119
2. Time required (including loading and assembly of caulking equipment)	1 hour 10 minutes	2 hours 15 minutes
3. Caulking compound used	Proprietary knife grade. Code 1A1-k. Penetration approx. 150 (25 g. 5 sec. 25°C.)	Proprietary heavy-bodied grade. Code 10C2-hk. Penetration approx. 60 (25g. 5 sec. 25°C.)
4. Number of joints caulked	7 full length 9 ft. 5¼ in.; 2 half length. Nominally ¼ in. by ⅝ in. cross-section	7 full length 9 ft. 5¼ in.; 2 half length. Nominally ¼ in. by ⅝ in. cross-section
5. Nominal capacity of joints	Approx. 163 cu. inch	Approx. 163 cu. inch
6. Quantity of material used	Approx. 120 cu. inch	—
7. Effective volume of gun	30 cu. inch	—
8. Effective volume of filler	265 cu. inch	—
9. Number of gun loads required.	4	—
10. Operating pressure	150 p.s.i.	—
11. Volume of gas expended (free air basis)	10 cu. ft.	—
12. Cost of gas expended. C'th Ind. Gases ruling price —air 5s. per 100 cu. ft.	6d.	—
13. Cost of labour (say, 4s. per hour)	4s. 8d.	9s.

5. Conclusion

In the trials reported in the preceding paragraph, use of the caulking gun enabled a saving to be made in labour to the extent of 4s. 4d. per house; the net saving being 3s. 10d. per house. This margin would, of course, be greater in "V.H.C." type concrete house designs calling for a larger number of joints, or in the several other prefabricated systems in existence where the outside wall is assembled from a larger number of narrow wall panels. In addition, caulking by this method greatly simplifies the application of more adhesive mastics. These, while frequently to be preferred on account of this adhesiveness, have on occasions been rejected on the grounds of manipulative difficulties.

NOTES

Meteorological Research

Although for many years systematic meteorologists have studied the day to day changes in Australian weather, studies of the fundamental physical phenomena underlying these changes have rarely been undertaken in Australia. The importance of the weather to Australia undoubtedly justifies a much more intensive study of the physics of the atmosphere; only a knowledge of the basic physical phenomena can ultimately lead to the possibilities of making successful long-range weather forecasts or of influencing such things as rain precipitation, and the Council has recently established a Section of Meteorological Physics Research to work in this field.

Mr. C. H. B. Priestley has been appointed as Officer-in-Charge of this Section. Since his arrival in Australia he has been engaged in surveying the many problems involved and in selecting a main line of work. It is proposed that an examination of the micro-structure of the atmosphere shall form the basis of the main line of work of the Section since it offers both a field for fundamental work and, subsequently, a wide variety of applications covering the whole range of meteorological problems.

It has been decided that the headquarters of the Meteorological Physics Section shall be in Melbourne, and initially approval has been given for the recruitment of a meteorologist (physicist or engineer by training) for the instrumental side of the main line of work, a meteorologist (analyst) with a good knowledge of charting methods and modern analytical techniques for the investigation of general and local circulations, and a theoretical meteorologist (mathematician) for work in dynamic meteorology and possibly also for the theory of turbulence.

North Australian Survey

The Commonwealth Government and the Governments of Queensland and Western Australia have established a North Australian Development Committee to examine and initiate proposals which will assist in the development of the northern areas of Australia. The Committee has recommended that C.S.I.R. should undertake a reconnaissance survey of the vegetation and soils of certain selected areas, and that, initially, the Darwin-Katherine region should be surveyed, followed by a survey of the Barkly Tablelands.

The survey party, under the leadership of Mr. C. S. Christian, of the Division of Plant Industry, comprised eleven members and has completed its work on the Darwin-Katherine region. The party is now embarking on the survey of the Barkly Tablelands. This is planned to cover 1947-48 and 1948-49, the party remaining in the field for some three months only in each year, returning to its headquarters for the remainder of the year for the purpose of recording data, identifying specimens, and compiling reports.

Future developments in the north must depend primarily on the extent to which pastures can be improved by the introduction and exploitation of pasture legumes and improved grasses, and experimental farms are being developed at Katherine, Northern Territory, and in the Ord River region in Western Australia to carry out investigations on soil, crop, and pasture problems.

Review

"THE FORAGE RESOURCES OF LATIN AMERICA: PERU,"

by Hugo W. Alberts.

(Bulletin 37 of the Imperial Bureau of Pastures and Forage Crops, 1947, pp. 24. Price 2s. 6d. Obtainable from the Central Sales Branch, Imperial Agricultural Bureaux, Penglais, Aberystwyth, Great Britain.)

A great deal of information on the plants and plant life of Peru has recently become available in the English language. The scholarly "Flora of Peru" by Macbride (Field Museum of Natural History, Botanical Series, Volume 13, 1936—), which is still in course of publication, gives a detailed account of the plants of the region, while the vegetation is admirably described in the introductory chapters by Dr. A. Weberbauer. Shorter accounts of the vegetation and plant resources are given by Williams and Hodge, respectively, in the volume on "Plants and Plant Science in Latin America," published by the Chronica Botanica Company.

In the Bulletin under review, the author follows the generally accepted division of the country into three main physiographic zones —Coast, Sierra, and Oriente—and for each of these zones describes the grazing animals, pastures, forage supplements, poisonous plants, and pasture management. Lucerne is the predominant pasture plant on the irrigated areas of the coastal region and in some of the valleys of the Sierra, and it is noteworthy that some of the Peruvian varieties are outstandingly productive, though usually short-lived in southern Australia. Native grasses, mostly of rather low value, form the bulk of the mountain pastures, and are grazed principally by sheep, alpacas, and llamas. Native legumes are apparently of little importance either in the Sierra or Oriente, but it would be of interest to know more about the grazing value of the 84 species of *Lupinus*, 26 of *Desmodium,* and 5 of *Stylosanthes* described by Macbride. One of the latter has shown outstanding promise as a pasture plant in northern Australia.

The Bulletin includes a map showing the physiographic zones, summaries in English, Spanish, and Portuguese, and a full index of genera and species. It is the second of a series dealing with the forage resources of Latin America, and both in form and content is well up to the high standard established by previous publications of the Aberystwyth Bureau.

W. HARTLEY.

Abstracts of Forthcoming Publications

" THE FRICTIONAL PROPERTIES OF WOOL AND OTHER TEXTILE FIBRES,"
by E. H. Mercer, B.Sc., and K. Rachel Makinson, B.A. To be
published in *J. Text. Inst.*

A stick-slip apparatus suitable for the study of the frictional
properties of textile fibres is described in detail, and a survey is made
of the results obtained with it.

A table is given of typical values of the coefficients of static friction
of various air-dry fibres and filaments. This coefficient increases as
the load decreases, the variation becoming rapid at loads less than
$0 \cdot 10$ g. wt. Possible causes of the effect are discussed.

For wool fibres rubbed on horn, two coefficients of static friction,
with-scale (μ_1) and against-scale (μ_2), are studied as functions of
pH and temperature. The nature and degree of contamination of the
fibre surfaces has a greater effect on the frictional properties than has
previously been realized. Variations in the degree of contamination
cause irreproducibility in the values of the coefficients of static friction.
A method of cleaning the fibres and maintaining them in a constant
condition, so as to obtain reproducible results, is described, and
the frictional behaviour of these fibres in the presence of
buffer solutions is compared with that of greasy fibres. The
frictional difference, $\mu_2-\mu_1$, is greater for greasy than for clean fibres.
For clean fibres the frictional difference is a maximum in the neigh-
bourhood of pH 7; this is in marked contrast with other published
results, which show that under less stringent conditions of cleanliness
the frictional difference is a minimum near pH 7.

The relation between the frictional properties and the handle of
wool is illustrated by measurements of the mean coefficient,
$(\mu_1 + \mu_2)/2$, for dry wool on horn. This coefficient is increased by
reagents which produce a harsher handle of the wool, and is decreased
by softeners.

The cause of the frictional difference of the wool fibre is discussed.
The increased frictional difference exhibited by greasy fibres can be
explained in terms of a ratchet effect of the cuticular scales, when the
full implications of such a mechanism are analysed. The observations
considered by other workers to be evidence against the ratchet theory
are shown to be compatible with it; it is concluded that the frictional
difference of wool is due to a ratchet effect.

" CONTRIBUTIONS TO THE STUDY OF THE CELL WALL. 4. THE NATURE
OF INTERCELLULAR ADHESION IN DE-LIGNIFIED TISSUES. 5. THE
OCCURRENCE, STRUCTURE, AND PROPERTIES OF CERTAIN CELL WALL
DEFORMATIONS," by A. B. Wardrop, M.Sc., and H. E. Dadswell,
D.Sc. Coun. Sci. Ind. Res. (Aust.), Bull. 221 (in press).

This Bulletin contains two further papers of the series on the cell
wall investigation which is being continued in the Section of Wood
Structure in the Division of Forests Products. The earlier papers of
the series were published in this *Journal,* Vol. 13, page 44 (Part 1),
page 129 (Part 2), page 290 (Part 3).

The results reported in Part 4 of the series provide some further
information on intercellular adhesion and tend to contradict an
earlier theory set out in Part 3 which postulated the existence of a

dilute-alkali-soluble, non-lignin, non-polyuronide bonding material which contributed to the holding together of the individual cells. This theory was challenged by Harlow of the New York State College of Forestry (*Paper Tr. J.* 112: 31, 1941) and additional investigations were therefore undertaken. It was necessary to determine whether cell for cell maceration did actually occur when delignified cross sections were treated with dilute alkali and the results observed microscopically. For this purpose such sections were, after treatment with the alkali, floated into water; this however did not cause cell for cell separation and it was found that the cells could be contracted into their original positions by means of alcohol. Similar results were obtained when phosphoric acid was used instead of the alkali. As no maceration took place it was concluded that the separation of the cells was due to the swelling of the cell wall although the alkali does take into solution something, presumably part of the cambial wall. Therefore the suggestion of a dilute-alkali-soluble, non-lignin, non-polyuronide material must be rejected. It is possible that the intercellular adhesion observed in delignified tissue is due to some mechanical adhesion between the cells.

Considerable attention has been paid by many workers to the nature and occurrence of slip planes and minute compression failures in the cell walls of wood. Part 5 of the series on cell wall studies deals in detail with these deformations and discusses their structure, properties, and the possible mode of their formation. It was apparent from the examination of numerous sections of both hardwoods and softwoods that slip planes are of common occurrence in nearly all wood fibres and tracheids. They have definitely been observed in the cell walls of fibres newly differentiated from the cambium. On the other hand they were not observed in compression wood tracheids. The occurrence of minute compression failures in Australian woods has received considerable attention because it has been abundantly demonstrated that they are characteristic of "brittle heart." Using the "broken fibre" test (broken fibres are found to be extremely common in macerated material taken from areas containing minute compression failures) as the criterion for the presence of "brittle heart" it has been shown that this defect occurs towards the centre of many trees and may extend on all sides of the pith; in some cases the distribution is eccentric. It has also been shown that "brittle heart" tends to be more common in the lower portions of the stem although here again the distribution varies. One very important observation was the very close association between the occurrence of reaction wood in both hardwoods and softwoods and the occurrence of minute compression failures. In hardwoods the bands of tension wood were shown to contain very numerous minute compression failures; in the softwoods minute compression failures were observed in close proximity to the bands of compression wood and generally on the pith side.

The influence of the cell wall deformations, particularly minute compression failures, on the properties of wood and on the properties of isolated fibres makes it important to have some knowledge of their actual structure and behaviour under certain conditions. This was obtained from observations on their optical properties, their staining reactions, and their susceptibility to chemical attack. It was found

that the optical behaviour of slip planes and minute compression failures is fundamentally the same as that of dislocation marks in textile fibres. These optical properties are governed by the micellar arrangement existing within the cell wall, and therefore it is reasonable to assume the possibility of difference in micellar orientation in the regions of the deformations. This has been recognized by earlier workers, and using their method of microscopic examination, namely, between crossed nicols, it was shown that the deformations in cell walls of wood represent regions of distortion in the general micellar arrangement. It was possible to measure the actual angle of micellar displacement in the region of the failures in relation to the longitudinal fibre axis. The staining reactions of the deformations suggested that their formation was accompanied by the rupture of the lignin-cellulose interface and that the micellar distortion produced results in an increased micellar surface which itself determines the preferential staining reaction. Several experiments were carried out to determine the reason for the susceptibility of these formations to chemical attack. It will be recalled that acid maceration of wood with minute compression failures present gives numerous broken fibres, while the same treatment on normal wood results only in whole fibres. It was considered that the susceptibility of the failures to chemical attack could be followed readily by employing acetylation and subsequently using a solvent for cellulose acetate. By this means it was possible to obtain broken fibres where the break definitely occurred in the region of the cell wall deformation. It was concluded that cell wall composition was not the underlying reason for the susceptibility of the deformations to chemical attack. It has been suggested rather that the susceptibility to hydrolysis and to acetylation is governed (a) by the greater ease of penetration of the reactant molecules because of micellar arrangement and increased intermicellar spaces and (b) by an increased reaction rate because of the locally increased micellar surfaces.

From a consideration of the distribution of these features in the growing tree it has been suggested that their formation is dependent mainly on the operation of growth stresses during the development of the tree. This suggestion is supported by the fact that minute compression failures can be artificially produced in the cell walls of timbers subjected to long-time loading tests. It has been concluded that while slip planes and minute compression failures are essentially similar in form they are not to be regarded as successive stages in failure under compression.

" Mechanical Composition of Soil in Relation to Field Descriptions of Texture," by T. J. Marshall, M.Agr.Sc., Ph.D.   Coun. Sci. Ind. Res. (Aust.), Bull. 224 (in press).

A triangular texture diagram has been drawn for the International fraction sizes (with limiting diameters of 2μ, 20μ, and 2 mm. respectively for the clay, silt, and sand fractions). This is based on scatter diagrams showing the relation between mechanical composition and field descriptions of texture made by the Division of Soils between 1939 and 1945. No serious departures have been made from standards already established in 1934 on the International basis.

It has been found from field descriptions that the relative amounts of sand and silt affect the apparent " clayiness " of a sample. Evidence is presented to show that limiting lines for the clay content of the texture classes should slope upwards towards the zero sand line in a triangular diagram. A theoretical basis is discussed for this departure from the practice followed in existing diagrams. Rearrangement on the basis of sloping lines has brought out a more logical relationship between classes than was apparent in the 1934 diagram. The new triangular diagram is compared with texture standards of the United States Department of Agriculture based on limiting diameters of 2 and 50μ respectively for the clay and silt fractions. Serious differences in the limiting percentages of clay for sandy loams, sandy clay loams, and sandy clays are noted and are discussed in the light of the sloping lines. An approximate adaptation is made of the new diagram (International fractions) to the fraction sizes of the United States Department of Agriculture.

A new type of texture diagram is presented in which the median size of the non-clay fraction is plotted against the clay content. The diagram is subdivided horizontally into categories of " fineness " of the non-clay fraction and vertically into categories of " clayiness." The diagram is unaffected by the size chosen for dividing the silt and sand fractions as is the case in the triangular representation of mechanical composition. Further, it allows differences due to the relative amounts of coarse and fine sand to be brought out. These two fractions have to be combined as total sand in the usual triangular diagram. Possible applications of this type of diagram are discussed. Symbols combining the " fineness " and " clayiness " categories are used to name the classes. These symbols avoid the confusion between fraction and texture names and the prejudice involved in the use of such terms as " loam." Conventional texture names are also applied to the diagram.

Descriptive standards have in general been maintained since the original examination was made in 1934. The chief departures in field practice are discussed. Methods are suggested for checking personal factors and for allowing for the effect of physical properties other than mechanical composition. The upper limiting size of the silt fraction has been reviewed and it was concluded that in Australian field practice, particles between 20 and 50μ in diameter are considered to be sand rather than silt.

Recent Publications of the Council

Since the last issue of this *Journal*, the following publications of the Council have been issued:—

Bulletin No. 200.—" Preparation of Core Ingredients for Searchlight Carbons," by T. R. Scott, M.Sc.

The " high intensity " arc used in modern searchlights requires a special type of positive carbon which has a central core consisting generally of a mixture of powdered carbon and fluorides of the cerium group of metals. When the arc is struck, with currents of 100-200 amps,

almost the whole of the flame is confined to the crater which forms at the end of the positive carbon, thereby producing a source of light much more intense than that obtained with the ordinary carbon arc.

Materials for the production of cored carbons in Australia have hitherto been imported, but the beach sands of northern New South Wales and southern Queensland contain considerable quantities of the mineral monazite, from which cerium fluoride can be prepared. In the present Bulletin, therefore, methods for the preparation of cerium fluoride from Australian monazite are given in detail, little information on this subject being available from the technical literature. A description is also given of the characteristics of the high intensity arc and of methods for the preparation and testing of cored carbons.

Samples of the Australian material were used to prepare cored carbons, which were tested in a searchlight arc lamp. They were found to be in no way inferior to imported cerium fluoride, even with considerable variation in the purity of the products when prepared by different methods. Some consideration was also given to the possibility of preparing core ingredients superior to cerium fluorides, but it was concluded that such an advance would probably not be made until more was known about the theory of the high intensity arc.

Bulletin No. 201.—" Grazing Management: Continuous and Rotational Grazing by Merino Sheep. 1. A Study of the Production of a Sown Pasture in the Australian Capital Territory under Three Systems of Grazing Management," by R. M. Moore, B.Sc.Agr., Nancy Barrie, B.Sc. Agr., and E. H. Kipps, B.Sc. *Appendix,* " The Measurement of Pasture Yield under Grazing," by G. A. McIntyre, B.Sc. " 2. The Effect of Continuous and Rotational Grazing on the Infestation of Sheep with Internal Parasites," by H. McL. Gordon, B.V.Sc., and Helen Newton Turner, B.Arch. 3. " Note on Pasture Management," by J. Griffiths Davies, B.Sc., Ph..D.

In the trial described in this Bulletin, a pasture mixture of *Phalaris tuberosa,* subterranean clover, lucerne, and cocksfoot, established in 1939 at Canberra, was grazed by Merino wethers from June, 1940, to October, 1944, according to three systems of grazing: continuous, 4-week rotation, and 8-week rotation. Comparisons were made of the yield and composition of the pasture, and the live weight, wool production, and health of the grazing sheep.

The grazing method had no effect on the yield of *Phalaris* or subterranean clover, but lucerne had disappeared almost completely after four years under continuous grazing. The yield of lucerne was limited under the 4-week rotation, but a productive stand was maintained under the 8-week rotation.

It was concluded that rotational grazing can only be expected to give increases in yield under certain severe conditions and that pasture management would be simplified if lucerne were grown as a separate crop—for hay or grazing.

Bulletin No. 205.—" Studies on the Breeding Performance of Ewes," by R. B. Kelley, D.V.Sc.

Since 1935 a series of studies has been made of relative fertility of ewes, particularly Merinos. In the trials described in the present Bulletin, Merinos were found to reach sexual maturity later than other

breeds. They did not attain their highest level of fertility until they were at least two years, and sometimes three or four years of age. Reduction in fertility occurred after the ewes were about eight years old. The delayed maturity was found to be associated with increasing rates of infantile mortality and incidence of death among ewes bearing first lambs.

On the other hand, cross-bred (Merino X Border Leicester) ewes showed a relatively high level of fertility at 21 or 22 months and appeared to reach the threshold of sexual maturity at about seventeen months. No information has yet been obtained on the effect of age on fertility in these ewes, since they have only been under observation for about six years.

Investigations are being continued on the problem of delayed maturity. So far, it is not known how widely spread in Merinos the condition may be.

Bulletin No. 208.—" Surface Fumigation of Insect Infestations in Bulk-Wheat Depots," by Frank Wilson and A. T. Mills.

This Bulletin discusses the fumigation of insects in the surface layers of wheat in Victorian bulk depots. The two principal insects concerned are the lesser grain borer (*Rhizopertha dominica*) and the long-headed flour beetle (*Latheticus oryzae*). The most satisfactory fumigant tested was carbon bisulphide, which gave an almost complete kill of *Rhizopertha* and satisfactory control of other insects when poured onto the surface of the wheat at a dosage of 16 fl. oz. per square yard. However, it must be used with great care to avoid explosions and health hazards.

A mixture of ethylene dichloride and trichlorethylene (3 : 1), when used at 45 fl. oz. per square yard, gives almost as good control as carbon bisulphide, and, though it is more expensive, it is much safer to use.

Fumigation of the insects in the top 2 feet of the wheat is sufficient, because in the deeper layers conditions are unsuitable for their development. The method was used in combination with mineral dusts to protect 3½ million bushels of wheat in the No. 1 depot at Murtoa, Victoria, and enabled it to be sold without price dockages after three years' storage.

Forthcoming Publications of the Council

At the present time, the following future publications of the Council are in the press:—

Bulletin No. 202.—" The Strain Complex and Symptom Variability of Tomato Spotted Wilt Virus," by D. O. Norris, M.Sc. (Agric.).

Bulletin No. 203.—" Agar in Australia," by E. J. Ferguson Wood, B.A., M.Sc.

Bulletin No. 204.—" A Soil Survey of Part of Waterhouse Estate, County of Dorset, North-East Coast, Tasmania," by G. D. Hubble, B.Agr.Sc.

Bulletin No. 206.—" Pedogenesis Following the Dissection of Lateritic Regions in Southern Australia," by C. G. Stephens, M.Sc.

Bulletin No. 207.—" The Fumigation of Wheat in Bag Stacks, by Frank Wilson and F. J. Gay, B.Sc., D.I.C.

Bulletin No. 209.—" Interaction of Surface Infestation, Temperature, and Moisture Content in Bulk-Depot Wheat," by Frank Wilson.

Bulletin No. 210.—" Preliminary Survey of the Natural Pastures of the New England District of New South Wales, and a General Discussion of Their Problems," by R. Roe, B.Sc. (Agric.).

Bulletin No. 211.—" The Water Retting of Flax," by W. L. Greenhill, M.E., Dip.Sc., and Jean F. Couchman, B.Sc.

Bulletin No. 212.—" The Frictional Properties of Lead-Base and Tin-Base Bearing Alloys: The Role of the Matrix and the Hard Particles," by D. Tabor, Ph.D.

Bulletin No. 213.—" Laboratory and Field Tests of Mosquito Repellents," by R. N. McCulloch, B.Sc., B.Sc.Agr., and D. F. Waterhouse, M.Sc.

Bulletin No. 214.—" The Preparation and Properties of Synthetic Cryolite," by P. Dixon, M.Sc., and T. R. Scott, M.Sc.

Bulletin No. 215.—" Studies in the Biology of the Skin and Fleece of Sheep. 4. The Hair Follicle Group and its Topographical Variations in the Skin of the Merino Foetus," by H. B. Carter, B.V.Sc., and Margaret H. Hardy, M.Sc.

Bulletin No. 216.—" An Examination of the Peet-Grady Method for the Evaluation of Household Fly Sprays," by D. F. Waterhouse, M.Sc.

Bulletin No. 217.—" The Relative Importance of Live Sheep and of Carrion as Breeding Grounds for the Australian Sheep Blowfly *Lucilia cuprina,*" by D. F. Waterhouse, M.Sc.

Bulletin No. 218.—" Studies of the Physiology and Toxicology of Blowflies. 12. The Toxicity of DDT as a Contact and Stomach Poison for Larvae of *Lucilia cuprina.* 13. Insectary Tests of Repellents for the Australian Sheep Blowfly," by D. F. Waterhouse, M.Sc.

Bulletin No. 219.—" Spray Tests against Adult Mosquitoes. 1. Laboratory Spray Tests with Culicine (*Culex fatigans*) Adults," by D. F. Waterhouse, M.Sc. " 2. Spray Tests with Anopheline (*Anopheles punctulatus farauti*) Adults," by D. F. Waterhouse, M.Sc., and D. O. Atherton, M.Sc.Agr.

Bulletin No. 220.—" The Preparation and Use of Harvey's Reduced Strychnine Reagent in Oceanographical Chemistry," by D. Rochford, B.Sc.

Bulletin No. 221.—" Contributions to the Study of the Cell Wall. 4. The Nature of Inter-Cellular Adhesion in Delignified Tissue. 5. The Occurrence, Structure, and Properties of Certain Cell Wall Deformations," by A. B. Wardrop, M.Sc., and H. E. Dadswell, D.Sc.

Bulletin No. 222.—" The Chaetognatha of South Eastern Australia," by J. M. Thomson, M.Sc.

Bulletin No. 223.—"Report of Marine Borer Survey in New Guinea Waters," by A. W. Shillinglaw, B.Sc., Dip.For., and D. D. Moore, B.Sc., A.S.T.C.

Bulletin No. 224.—"Mechanical Composition of Soil in Relation to Field Descriptions of Texture," by T. J. Marshall, M.Agr.Sc., Ph.D.

Bulletin No. 225.—"Studies on the Control of Wheat Insects by Dusts. 1. Field Tests of Various Mineral Dusts against Grain Weevils," by F. J. Gay, B.Sc., D.I.C., F. N. Ratcliffe, B.A., and R. N. McCulloch, B.Sc., B.Sc.Agr. " 2. Further Tests of Various Mineral Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C. " 3. The Use of Dust Barriers for the Control of Grain Insects," by F. J. Gay, B.Sc., D.I.C. " 4. The Use of DDT– and 666– impregnated Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C.

Bulletin No. 226.—" An Ecological Study of the Australian Plague Locust (*Chortoicetes terminifera* Walk.) in the Bogan-Macquarie Outbreak Area, N.S.W.," by L. R. Clark, M.Sc.

Bulletin No. 227.—" Studies on Perennial Veldt Grass (*Ehrharta calycina* Sm.)," by R. C. Rossiter, B.Sc.(Agric.).

" The Commercial Timbers of Australia—Their Properties and Uses," by I. H. Boas, M.Sc.

" Handbook of Australian Pelagic Tunicates," by Harold Thompson, M.A., D.Sc.

PLATE 1

Furfural· A Pilot Plant Investigation of Its Production from Australian Raw Materials (See page 225)
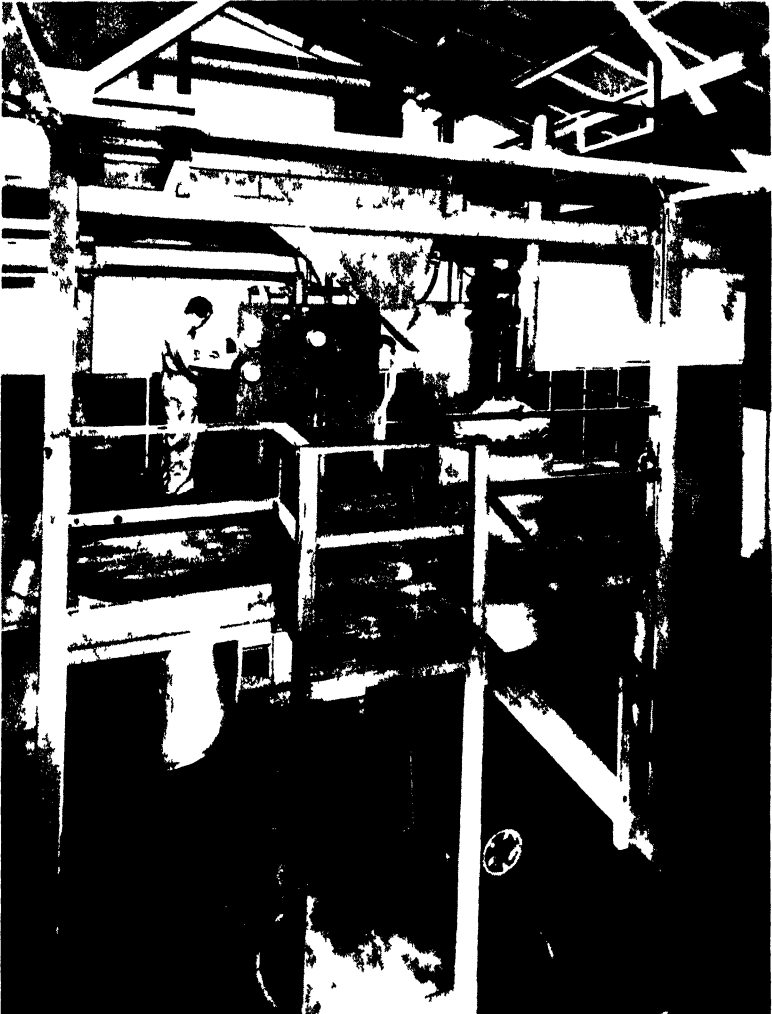
Furfural pilot plant digester and auxiliary equipment

PLATE 2

Electrolytic Polishing of Copper in Orthophosphoric Acid.
(See page 297.)
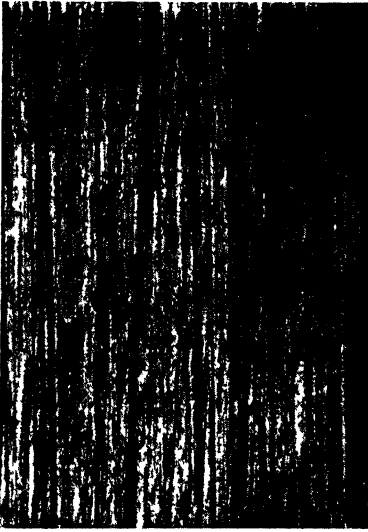
FIG 1 Copper specimen prior to polishing prepared on 600 carborundum paper × 200

FIG 2—Specimen prepared in the range AC The surface is deeply etched × 200.

FIG 3—Specimen polished on range DE The surface has been lightly etched to show up the grains The black spherical particles are copper oxide. × 200.

FIG 4 Specimen polished in range above E The presence of fine gas bubbles causes pits on the surface. × 200.

PLATE 3

A Pneumatically Operated Caulking Gun   (See page 306)
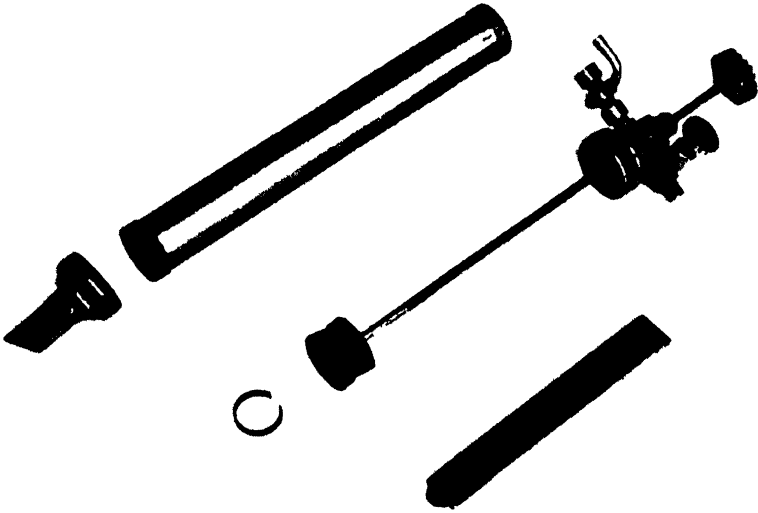
FIG 1   Pressure caulking gun dismantled

FIG 2—Operation of caulking gun   The gun is being used to caulk
a wall joint in a V H C precast concrete house

PLATE 4

A Pneumatically Operated Caulking Gun. (See page 306.)
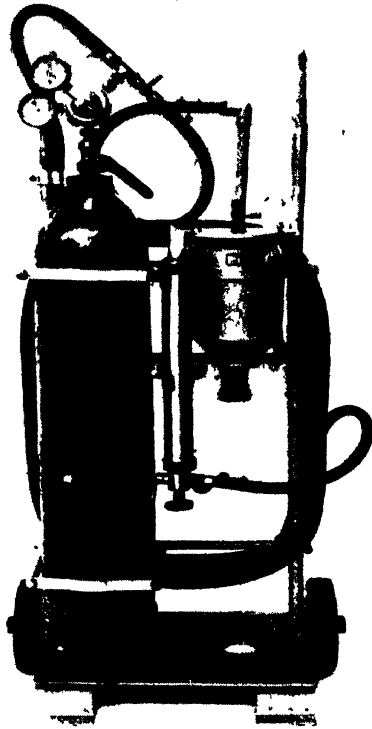
FIG. 1.—Pressure caulking equipment
mounted on hand trolley.
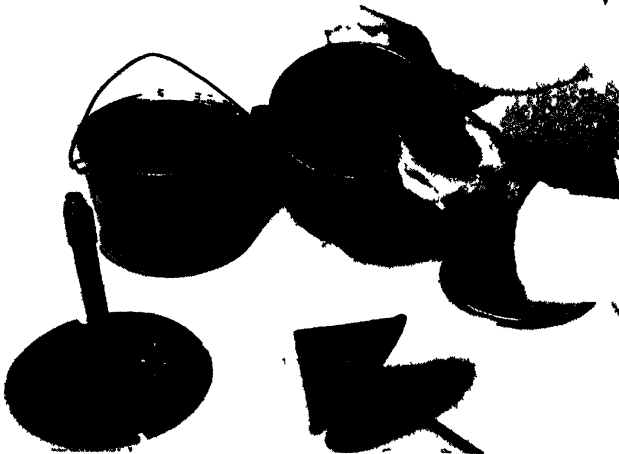
FIG. 2.—Loading filler with caulking
compound.

FLOW SHEET OF PLANT DESIGNED FOR COSTING FURFURAL PRODUCTION [FOR KEY SEE TABLE 8 (?3)]
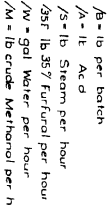
/b = lb per batch
/A = lt Acid
/S = lb Steam per hour
/3sf lb 35% Furfural per hour
/W = gal Water per hour
/M = lb crude Methanol per hour

8086 /Residue B
1830 /Dry

From ①

3235 lb oat husks /B

[/A 35/5 for 3 hrs]
[160 /S · 3 hrs]

606 /Sg

[/S 160 /4F for 3 hrs]
[/A 620 /4F for 1]

8090/4F B

6255/WB

8090/4F B

1883.4 F

266/S

200F/0.1F

477/S

/M

/5

/W

113/W

5/35F

27/35F

32.45F

24F

70/95F

55/W

81/84F

359/W

318/35F

348.35F

427.7H

9.4F

/18.4

57.6F

66.7/99.95F

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The Extension of the Card-Sorting Method to War-time Problems in Timber Identification

By H. E. Dadswell, D.Sc.,* Audrey M. Eckersley, M.Sc.,* Florence V. Griffin,* and H. D. Ingle, B.For.Sc.*

Summary.

The war-time activities of the Division of Forest Products with respect to the development of simple means for the identification of the timbers in the various Pacific operational areas have been described.

For the benefit of other workers in this field a detailed description has been given of (a) the first sample card-sorting key developed for the identification of timbers of the South-West Pacific area, and (b) the modified and finally adopted card-sorting key for New Guinea timbers.

Reference has also been made to the development of similar keys for (a) the timbers of the Philippines, and (b) the timbers of Malaya and the East Indies.

Various factors in this type of identification key have been discussed and reference made to its value for field identification.

1. Introduction

For some time after the outbreak of hostilities in the Pacific little or no attention was given to timber identification problems. Then, suddenly it was realized by everyone that timber, because of its universal usefulness, was still a most valuable munition of war. This being so, it was often essential to know the identity of the various timbers being used, so that their use could be to the best advantage. While the problem was not particularly serious when bases were being built in Australia, mainly because the properties and identity of the more common Australian timbers were fairly well known, it did become serious when large bases were built in the islands to the north of Australia where local timbers had to be used because of the lack of sufficient shipping to bring timber from Australia or from North America. It was at this stage that the Division of Forest Products was asked to assist in (a) providing information regarding the properties and uses of all island timbers and (b) suggesting methods by means of which such timbers could be identified as accurately as possible by the personnel of appropriate units in the various Services. It was immediately obvious that these demands could probably be met satisfactorily by the development of some simple type of

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* An officer of the Division of Forest Products.

one region, and consideration was therefore given to the possibilities of a key of the card-sorting type which would have the added advantage that information regarding any particular timber could be included on the card for that timber.

The application of the card-sorting principle to timber identification problems had been well established as early as 1936, and has been used in various laboratories where timber identification is carried out. Clarke (1938) published details of a perforated card key for identification key covering the more important timber species of any hardwood timbers, and Phillips (1941) described a similar type of key for the identification of coniferous timbers. The principle was further extended by Dadswell and Eckersley (1941) to a rather more specialized problem, namely the identification of the various timber species of the genus *Eucalyptus*. In all these keys, however, the emphasis has been on identification in the laboratory, where examination of details of wood anatomy can be completed at leisure, using thin sections and the microscope. The Forest Products Laboratory, Princes Risborough, England, had, however, proposed a card-sorting key based on the use of the hand lens only and details were set out in a Progress Report which the authors have had the privilege of consulting.

The Section of Wood Structure of the Division of Forest Products has since 1936 employed the card-sorting method for identification purposes; two systems, one for hardwoods and the other for coniferous woods, were developed, in addition to the specialized key for the eucalypt timbers previously referred to. These have proved their value many times over and duplicate sets have been forwarded to the Australian Forestry School, Canberra, and the various State Forest Services. Again, however, these keys were designed for laboratory use by wood anatomists, and, invaluable as they are, they are not the type for field use by operators with little or no knowledge of wood anatomy. The problem therefore was the development of a simple card-sorting key using macroscopic features only, so that any one without specialized training in wood technology could operate it. The latter point was essential because it was visualized that such a key would be used by engineer and forestry units in both the Australian and American Armies.

In as much as New Guinea was for some time the centre of action for the Pacific war, the first attempt at the development of a simple card-sorting key was made using a number of the more important New Guinea timbers. Information on these timbers was obtained from examination of available material of the species selected (50 in all), many of which occur in other areas of the South-West Pacific as well as New Guinea. Thus it was fortunate that the Division had in its possession collections of various timbers from New Guinea, the Dutch East Indies, Malaya, and the Philippines. In addition some authentic material was available from New Guinea, and Mr. C. E. Lane-Poole, then Inspector-General of Forests, made available pieces of timber collected by him during his survey of New Guinea (Lane-Poole, 1925). This first key, described in some detail below, was perhaps over-simplified in that it was based on macroscopic features visible to the naked eye only (i.e. a hand lens was neither an essential

nor a desirable aid). The cards for the 50 selected New Guinea timbers, each suitably notched at the perforations corresponding to the features observed for each species, together with various supplementary sheets of instructions, definitions, &c., were issued in a suitably prepared cardboard container. A number of such sets were despatched to Australian and American Army Units—mainly Engineers. The key proved very successful even when used by untrained personnel. However, with the transfer to New Guinea of various Australian Forestry Units and as a result of further work, it was felt that a larger, more detailed key should be developed. In this second key more timbers were included and a wider range of features employed, including those visible with a hand lens, for it was considered that the personnel of forestry units would have sufficient training to use a hand lens key of this type (described in detail below). In addition, arrangements were made by the Australian Army through the Engineer-in-Chief for one of us (H.E.D.) to visit forward areas to give instructions on the correct methods of using the key. It was indeed surprising how rapidly persons unacquainted with wood technology mastered the procedure of identification by this means and how effectively they could use the key. While in New Guinea with the Australian Army Unit, New Guinea Forests, additional material of the various more or less known commercial timbers and of other species with potential commercial value was collected. Examination of this material indicated the need for modifications and additions, and eventually the key for the timbers of New Guinea and neighbouring islands covered some 140 different species. As a result of the demand for information on uses and properties of each of these, provision was made on the cards for strength grouping, durability classes, and possible uses.

Concurrently with the extension of the key for timbers of the South-West Pacific area, the Division was busil; engaged in the preparation of a similar type of key for the commercial timbers of the Philippines. The request for 300 sets of such a key was received through the Allied Geographical Section of the South-West Pacific Command, who demanded 150 of these keys before the invasion of the Philippines. This demand was met, even though it was requested that the card for each species include botanical information and where possible illustrations of botanical features. Again the cards were adapted so that sortings could be made on properties and uses as well as on anatomical features. Most of the information on the timbers was gathered from the publication on " Philippine Woods " by Luis Reyes (1938); other information was gained from the examination of available timber specimens.

The next request came from the Australian Army, and card-sorting keys covering timbers of Borneo were urgently required. Because many of the important timbers of this region were also common in the other islands of the East Indies and in the Malay Peninsula, a comprehensive key covering timbers (117 in all) of Borneo, Java, Sumatra, Malaya, and Burma was developed following the lines adopted in the case of the Philippine timbers. Before the invasion of Borneo 100 sets were prepared and 25 forwarded to the Australian Army through the Engineer-in-Chief's Branch. Sixty sets were sent to the South-East Asia Command through the Allied Geographical

Section, South-West Pacific Command. Once again botanical information on the various species was included, together with information on the properties and uses of the timbers.

Special card-sorting identification keys covering (*a*) timbers of Northern Territory and (*b*) Australian and Island timbers used for marine piling, were also developed for use by Australian Army personnel. In all, in less than twelve months, over 60,000 individual cards were prepared and, as most of the work on notching, affixing photographs, &c., was done by hand, it will be appreciated that a considerable effort was involved.

2. Description of the First Identification Key for Timbers of the South-West Pacific

The cards adopted for this first key were 5 in. by 4 in. in size with perforations along both 5 in. sides. Forty of these perforations were available and of these, 39 were allotted to the possible variations in macroscopic features as shown in Fig. 1 and as listed below. The features were selected so as to give a fairly wide range of variation in weight, colour, pore size, and arrangement, vessel contents, rays, and soft tissue, in addition perforations were allotted to locality and topography. For every timber included the descriptions and notchings for positive features were based on the macroscopic examination of

SPECIES **Anisoptera polyandra.**

KNOWN NATIVE NAMES garawa(Buna), karawa (Binandele) karalaka (Vailala).

Fig. 1.—Face view of card used in the first card sorting key for timbers of the South-West Pacific Area—macroscopic features used only.

numerous specimens of the wood (without use of hand lens). Thus the pore size and arrangement recorded were based on the examination of a cleanly cut cross section, as was the description of soft tissue and rays. On the front of the card the botanical name of the timber was typed together with known native names and/or the name by which the timber is commonly known. On the back of the card for each species was pasted a photograph of the cleanly cut end section of the wood taken at approximately three magnifications; this gave a very good picture of the arrangement and size of pores, soft tissue and rays. Also on the back of the card, details of the timber, its colour, density, durability, uses, and special properties were given (see Fig. 2). For the benefit of those untrained in wood technology and lacking knowledge of the various terms used, a number of definitions were included with the prepared cards together with detailed notes on how to use the key and explanations of the reasons for using various features. Further, a photographic print of a series of diagrams illustrating various structural features was prepared and issued with each batch of cards.

Remarks: Wood pale yellowish brown. Vertical canals shew white deposits & may be in concentric bands or scattered among pores. Timber useful for building construction(scantling). A.D.density approx. 40 lb./cu.ft

Fig. 2.--Back of card shown in Fig. 1.

It has been considered desirable in this brief review of the work to include some reference to the various features used, and to the underlying reasons for the choice of these features. This information may prove of value to other workers desirous of developing indentification keys of this type. The details are set out below:—

General Heading.	Feature Number.	Feature Description.	Remarks.
Location	1 2 3 4	New Guinea .. .. New Britain and neighbouring islands .. Dutch East Indies .. Philippines	Some indication of the known distribution of each species is valuable
Topography ..	5	Highlands ..	Areas over 4,000 feet above sea level
	6	Foothills	Areas from 1,000 to 4,000 feet above sea level
	7	Coastal	Areas up to 1,000 feet above sea level
Weight .	8 9 10	Floats easily . . Barely floats .. Sinks . ..	There are difficulties in assessing the density of any timber especially in the green state, hence the three suggested weight variations
Colour	11 12 13	White, pale brown, pale yellow Pink, red Other colours .	Classification on a colour basis is not easy, therefore two main groupings were used to cover the pale timbers and the red timbers Under other colours it was intended to include bright yellow, dark brown, chocolate, and any variegated colours
Others features ..	14	Odour .	Many timbers have a characteristic odour
	15	Frothing test positive	This refers to the fact that shavings from some timbers froth when shaken with water
	16	Vertical canals	Designed to cover the canals of the Dipterocarpaceae as well as the anomalous type observed in some other species
	17	Ray figure conspicuous	Many timbers have a conspicuous ray figure or ray fleck on split or sawn radial surfaces
	18	Ripple marks ..	Storied structure as seen on a freshly cut tangential surface

General Heading.	Feature Number	Feature Description	Remarks.
Others features—*continued.*	19	Hard to cut across grain	Test carried out with pocket knife; designed to pick out the very hard and the very soft timbers
	20	Very soft to cut across grain	
Pore size (appearance in cross-section)	21	Large ..	Readily seen with naked eye
	22	Very small	Indistinct or invisible to naked eye
	23	Intermediate in size	Just visible to naked eye
	24	Absent	To cover all coniferous timbers
Pore arrangement and contents (appearance in cross section)	25	Solitary .. .	More than 75 per cent. solitary as in various eucalypts
	26	Oblique chains	
	27	Tangential arrangement	As in members of the Proteaceae
	28	Radial arrangement	The typical radial multiples
	29	Clusters	
	30	Ring-porous	
	31	Contents conspicuous	To cover the white or coloured contents of vessels and tyloses
Soft tissue (appearance in cross-section)	32	Not visible to naked eye	This also covered "absent"
	33	In regular* bands wider than pores	The width of the bands in comparison with the radial diameter of pores
	34	In regular* bands narrower than pores	
	35	In irregular* bands	E g , terminal
	36	Wing-like	
	37	Surrounding pores	
Rays (appearance in cross-section)	38	Equal to or wider than pores	I.e., in comparison with average tangential diameter of pores
	39	Narrower than pores	

* "Regular" and "irregular" designed to cover the radial spacing of the soft tissue band

A series of simple notes was prepared for the guidance of users of this key and certain of them are of general interest, namely:—

(i) Where a red line was placed opposite the notch for any particular feature it meant that this feature was not readily discernible to the naked eye, although visible with a hand lens.

(ii) Where a black line was placed opposite the notch for any particular feature it meant that the feature was not common for the timber in question.

(iii) Stress was laid on the desirability of sorting on the most obvious features first, with no reference to the order of appearance on the cards—doubtful features to be left or considered only at the end.

(iv) Only a limited number of the most important timbers (50) were included in the key; in any area other timbers not included in the key might be encountered.

(v) Details of structure are more clearly revealed if the cleanly-cut cross-sectional surface is moistened with water.

(vi) In a macroscopic key of this nature it is rare for only one card to remain after a number of sortings. When several cards remain reference should be made to the photographs pasted on the backs of each card and to the details of colour and properties given thereon as a guide to final identification.

With the above information it should be possible to use the key effectively for those timbers covered by it. This was proved in the laboratory where untrained juniors using the key made a high percentage of correct identifications, and in the field where in many cases quite untrained operators effected satisfactory identifications. However, as intimated earlier, it was soon felt that the card-sorting type of key would be much more effective if more species were covered and if use was made of a hand lens in the examination of the wood.

3. Description of the Enlarged and Modified Identification Key for Timbers of New Guinea and Neighbouring Islands

In this key the size of the card was increased to 6⅜ in. by 4 9/16 in. and it was perforated on all edges. On the face of the card there were 60 perforations available for use, as shown in Fig. 3. In the later models twenty spare perforations on the narrow edges were utilized for strength groupings, durability classes and uses, as shown in Fig. 4, by using the back of the card. Thus a total of 80 perforations was available for notching if and when required. It will be seen that from the identification angle 21 additional perforations were available for coding to possible variations in macroscopic features in comparison with the first card sorting set described above. More attention was given to variations in colour, weight, hardness, and because of the greater detail visible with the hand lens, more variations in ray structure, soft tissue, and pore size and arrangement were utilized. The final form of the cards for this key was not completed until early 1946, but by June, 1944, the first sets, with the 60 possible variations as shown in Fig. 1, were forwarded to operational areas. Subsequent modifications were mainly concerned with:—

   (a) The addition of information on the strength groupings, durability classes, and uses as mentioned above.

   (c) The addition of new species which has increased the present size of the set to a total of 137 species.

,   (b) **Revision of the species investigated earlier as a result of additional material becoming available for examination.**

On the back of each card was pasted a low-power (3×) photograph of the cross-section of the timber described. The cards were issued in a suitable container (first sets cardboard, later wood) and were accompanied by a colour chart for assistance in matching colours, a booklet of explanatory notes, a photographic copy of illustrations of various characteristic anatomical features, and a needle for sorting purposes.

FIG. 3.—Face view of card finally adopted for use in card sorting key for New Guinea timbers. Macroscopic features visible with hand lens used.

It was suggested that a piece of timber to be identified should be prepared and examined according to the following procedure:—

- (*a*) Make a clean cut on the cross-section using a very sharp knife or razor blade.

- (*b*) Examine the cleanly cut cross-section using a hand lens, noting direction and size of rays; number, size, arrangement, and contents of pores; arrangements of soft tissue.

- (*c*) Split along true radial and tangential surfaces and clean up the tangential surface with a knife or a razor blade.

- (*d*) Examine radial surface for marked difference in colour between rays and background.

- (*e*) Examine tangential surface for ripple marks and presence of gum canals.

- (*f*) Note colour, impression of hardness by cutting across grain, and approximate density, remembering that hardness and density are affected by moisture content.

- (*g*) Keep a look out for other features, such as definite odour, distinct oiliness, or greasiness; or stickiness, presence of concentric vertical canals, and ability of shavings to produce froth when shaken with water.

The anatomical features and physical properties used were similar to, but did not exactly follow, those given above in the description of the first macroscopic key. They are listed below with explanatory remarks:—

Colour.

It was considered of advantage to widen the colour groupings and, as colour classification can be very difficult, a colour chart was provided so that the operator might assess the colour of dry longitudinal surfaces of the specimens under examination. It was pointed out that colour will vary considerably depending on (*a*) moisture content, (*b*) sapwood or truewood, (*c*) age of tree from which specimen has been taken, (*d*) age of specimen, (*e*) the surface examined, (*f*) presence of decay or sap stain.

1. Whitish, pale-brown, pale-yellow, straw.
2. Dark-brown (a definite brown).
3. Pink or red tints, including red-brown.
4. Other colours, e.g. black, purple, orange, bright-yellow, &c.   i.e. any colour not specified under 1, 2, or 3.
5. Mottled or streaky.

Rays.

In this key there were no codings for heterogeneity or homogeneity, although with the hand lens it is often easy to pick on the upright ray cells and to detect oil cells on a split radial face. Reference to these, however, was usually made on the back of the cards.

6. Broad and conspicuous on cross-section	Self explanatory, similar to oak or the silky oaks.
7. As wide or wider than pores	Width of ray estimated by comparison with tangential diameter of pores—a useful feature as proved from first set.
8. Narrower than pores	
9. Not clearly visible even with lens	In some cases rays are too narrow or not of sufficient contrast to be seen on cross-section even with a hand lens.
10. Conspicuous on radial surface owing to colour	This is irrespective of size and meant to refer only to colour prominence; experience showed that it was not a particularly useful feature.
11. With gum canals	The canals appear under lens as dark cavities in rays and may be seen on freshly cut tangential surfaces or split radial surfaces. A good positive feature.

Soft Tissue (Wood Parenchyma).

The groupings in this section (soft tissue) were selected to give greatest possible assistance in sorting. In the preparation of the individual cards it was sometimes necessary to notch more than one perforation in this group to cover variation in the species or possible difficulties in interpretation. It was often particularly difficult to differentiate between " diffuse " and " reticulate."

12. Absent	To cover species on which soft tissue is absent or difficult to see under lens.
13. In regular bands wider than pores	Regular bands are those which occur at regular intervals as seen on cross-section. Width as in earlier key.
14. In regular bands narrower than pores	
15. In irregularly spaced bands ..	e.g terminal.
16. Surounding pores . ..	Difficult to detect even with lens.
17. Wing-like, confluent.	
18. Diffuse . .	Cells scattered irregularly.
19. Reticulate	In fine lines only visible with lens and forming a sort of network with the rays.

Other Features.

20. A definite odour . .	Remarks as for first key.
21. Frothing test positive ..	
22. Ripple marks .. .	
23. Vertical canals concentric ..	
24. Distinct oiliness or greasiness	An additional feature, detected by feeling longitudinal surfaces: stickiness of the surface is also allowed for here.

25.

Topography.

26. Highlands . .. ..|	As in first key
27. Foothills	
28. Coastal	
29. Swamps	Additional; intended to cover mangrove swamps and other swampy areas.

Locality.

30. Australia.
31. New Guinea and neighbouring| islands
32. Celebes, Timor, Moluccas
33. Borneo, Java, Sumatra
34. Philippines   . .
35. Malaya   . .   . .

Divisions made on somewhat different lines from those followed in first key.

Pore Number.

36. Few   . .   . .   4 or less per sq.mm.
37. Moderately numerous   5-11 per sq.mm.
38. Very numerous   . .   More than 11 per sq.mm.

The counting of the actual number of pores was accomplished by using one of the holes in the printed card (see Figs.). These holes were found to be approximately 7 sq.mm. in area and therefore the actual pore number per sq.mm. for any timber could be obtained simply by dividing by seven the number counted in the area outlined by one perforation when the card is pressed on to a cleanly cut end section of the wood. Counts should preferably be made over several different areas and the mean taken.

Pore Size.

39. Large   . .   . .   Large and separately distinct to naked eye.
40. Intermediate   . .   Visible to naked eye without any strain.
41. Small   . .   . .   Indistinct to naked eye.
42. Very small   . .   Indistinct even with hand lens.

Pore Arrangement (and Contents).

43. Absent   . .   . .
44. Ring-porous or semi ring-| porous

Remarks as in first key.

45. Predominantly solitary   . .
46. Radial multiples up to and including 4
47. Radial multiples over 4   . .

Considered of value to have separate perforations for long radial multiples.

48. Oblique   . .   . .   . .
49. Tangential   . .   . .
50. Clusters   . .   . .   . .

Remarks as in first key.

51. Tyloses common   .   . .

A separate perforation was taken for tyloses but should be used only when they are common enough to be quite distinct.

52. White or yellow deposits common

Again a separate perforation; the difference in appearance between white deposits and tyloses have been described.

Weight.

53. Very heavy

Above 62·5 lb./cu.ft. air dry to approximately 17 per cent. moisture content.

54. Heavy to moderately heavy . .   50-62·5 lb./cu.ft.
55. Light to moderately light or moderately heavy   30-49 lb./cu.ft.
56. Very light   . .   . .   . .   Less than 30 lb./cu.ft.

The whole weight range was included but it was considered that perforations 53 and 56 would prove the most useful.

Hardness.

This is correlated with weight, but in these keys refers mainly to the difficulty in cutting air-dry wood across grain with a pocket knife. It was thought that here again the extremes would be most useful.

57 Very hard and horny to cut
58 Hard to cut
59 Intermediate to cut
60 Very soft to cut

Strength Groups (see Langlands and Thomas, 1941)

63 Group A
64 Group B
65 Group C
66 Group D

The timbers were classified into four groups as is done for Australian timbers using the following average properties selected by the Section of Timber Mechanics:—

Group	Modulus of Rupture (lb /sq in)		Modulus of Elasticity (lb /sq in)		Crushing Strength Parallel to the Grain (lb /sq in)		Shear Strength (lb /sq in)	
	Green	12° m c	Green	12% m c	Green	12% m c	Green	12% m c
A	15,000	24 000	2,400,000	3,000,000	7,500	12,000	2,000	2,500
B	12,000	20 000	2,100,000	2,600,000	6,000	10,000	1,500	1,900
C	10,000	16,000	1,700 000	2,200,000	5 000	8,000	1 200	1,600
D	7,000	12 000	1,500,000	1,900,000	3,350	6,000	800	1,100

Note —The above figures apply to defect free timber only   If a card is not notched at one of the above perforations either no information on strength properties is available or it falls into a class below Group D   The latter alternative would certainly be the case with very low density timbers

Durability Classes (Truewood only).

68 Class 1
69 Class 2
70 Class 3
71 Class 4

Class 1 was meant to cover species which when used in exposed situations were highly durable, while class 4 covered those of low durability. This classification was designed to refer to attack by fungi and termites only, and not to resistance to mechanical wear, or to attack by marine organisms. The placing of the various species in these classes was somewhat uncertain because of the lack of detailed knowledge. However, it is a general indication.

Uses.   Features 74-85 inclusive   See Fig 4 for details

The inclusion of information on strength, durability and uses rendered the sets more generally useful, as the card for each species can be considered as giving the available information on properties,

uses, and structure for that species. The latest model of this key has definite post-war application and a number of sets have already been distributed.

74	75	76	77	78	79		80	81	82	83	84	85
PILING	POSTS	HEAVY CONSTRUCTION	LIGHT CONSTRUCTION	CASES	VENEERS		TOOLS AND HANDLES	BOAT DECKING AND PLANKING	BOAT FRAMING	TURNERY	FURNITURE	INTERIOR FINISHING
		USES							USES.			

Colour yellowish brown, concentric rings of vertical canals present (hence 15 Notched) and showing white deposits also in pores Pore number 7-9/sq mm A.D density 43 lb/cu ft Soft tissue barely visible, surrounding pores.

Microscopic features: Cells of vertical canals appear separated and about $\frac{1}{8}$th to 1/5th size of largest pores, rays heterogeneous 3-7 seriate, ray-vessel pits large, simple, rounded or oval, parenchyma paratracheal and extending in short uniseriate lines.

	STRENGTH GROUPS					DURABILITY CLASSES				
	A	B	C	D		1	2	3	4	
61	62	63	64	65	66	67	68	69	70	71	72	73

FIG. 4.—Back view of card shown in Fig. 3.

4. The Card-Sorting Key for Timbers of the Philippine Islands

This key was in essence identical with that prepared for the New Guinea timbers and described in detail in section 3 above. However, there were certain modifications and additions. On the face of each card was printed the specific name and common name of the timber, while on the back of the card there was affixed an annotated photograph of a cross-section of the timber magnified approximately three times together with a brief timber description, notes on the size and

type of tree, botanical characteristics in simplified terms (see Fig. 5), information on the occurrence of the species in the Philippines. In addition, where available, a line drawing of leaves, flowers, and fruit was included. Many of these diagrams were obtained through the courtesy of the National Herbarium, Melbourne. They were prepared from photographic copies of illustrations or from photographs of herbarium material. The details in such photographs were outlined with indian ink and the unwanted portions bleached out leaving suitable line drawings from which the blocks were made.
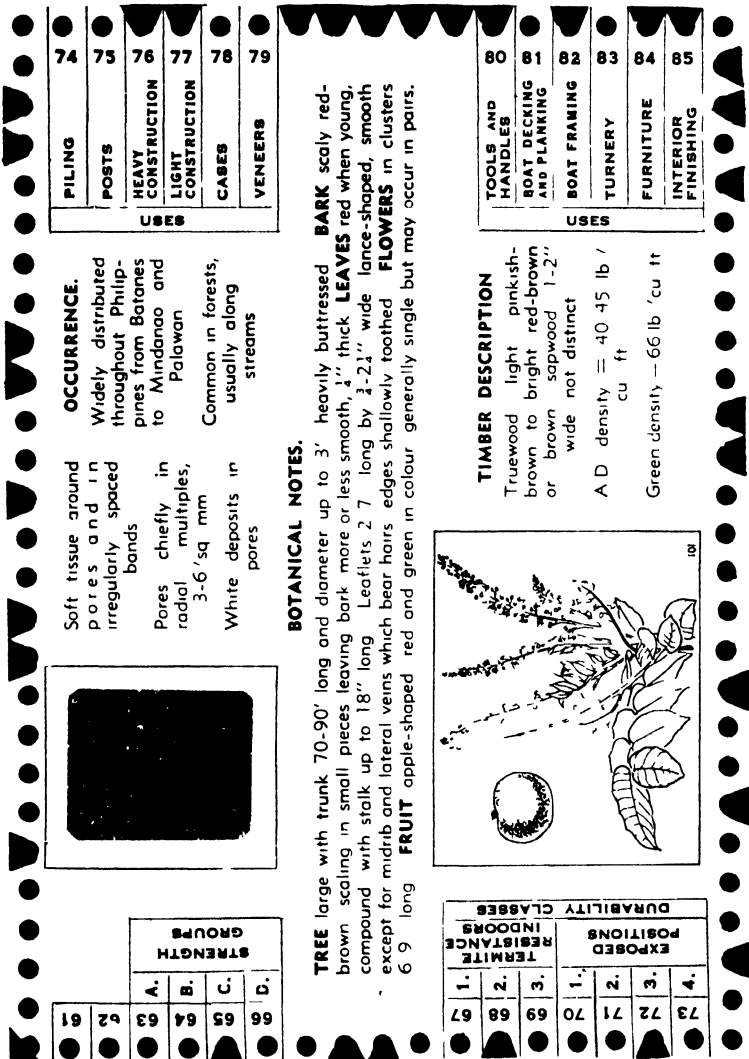
FIG. 5.—Back view of card adopted for use in card-sorting key for Philippine timbers.

Most of the information on the timbers was obtained from " Philippine Woods " by Luis Reyes and this covered the strength groupings, durability classes, and uses. For the first of these, the

data given by Reyes were used to place the various timbers in the strength groups employed by this Division and set out in detail above. For the durability classes Reyes's information was transposed directly and this meant using perforations 67-73 inclusive and subdividing as follows:—

Termite Resistance Indoors.

67. Reyes's class 1	.	Very resistant.
68. Reyes's class 2	..	Resistant to moderately resistant.
69. Reyes's class 3	..	Susceptible.

Durability Exposed Positions.

70. Reyes's class 1	..	Very durable—lasting over nine years.
71. Reyes's class 2	.	Durable—lasting 4½-9 years.
72. Reyes's class 3		Moderately durable—lasting 2½-4½ years.
73. Reyes's class 4 and 5	..	Perishable—lasting less than 2½ years.

It will be noted that these classifications are somewhat different from those employed for the New Guinea timbers but as it was intended that the cards be used in the Philippines it was considered desirable to retain the durability classes developed there.

Again the cards were accompanied by colour chart, booklet of instructions and explanatory notes, illustrations of various anatomical features, and a needle for sorting. All were housed in a neat wooden box with sliding lid.

5. The Card-Sorting Key for Timbers of Burma, Malaya, and the East Indies

The cards in this key were similar to those used for the Philippine timbers. The botanical and common name of each timber was printed on the face of the card for that timber while on the reverse side the illustrations of botanical features, botanical notes, timber description, and photograph of the cross-section of the wood were again included. Perforations were allotted to strength groups, durability classes, and uses as in the card for New Guinea timbers illustrated in Fig. 4. The information on the botanical side was obtained through the kind co-operation of the Director of the National Herbarium, Melbourne, and the Government Botanist, Brisbane, and his staff.

The information on the timbers of the various localities covered was obtained both from examination of authentic material from these localities and from published information on the various species selected for inclusion. The actual number of timbers (117) naturally does not cover to any extent the species occurring in these regions. They were selected for inclusion on the basis of properties, occurrence, and likelihood of use by Army engineers.

6. General

In the two other special keys prepared (referred to in the Introduction) the same type of card was used as for the final modified New Guinea set. Six sets were prepared of the key for the Northern Territory timbers and a total of 40 species covered. These sets were used by the Australian Army Forestry and Engineer Units in that area. Only two sets of the special key covering Australian and New

Guinea timbers suitable for marine piling were prepared. Forty-two species were covered. This key was used by Major A. W. Shillinglaw of the Engineer-in-Chief's Branch of the Australian Army in a special survey of piling carried out in the New Guinea operational zone (Shillinglaw and Moore, 1945).

In war-time it is, of course, difficult to find out just how effective something of this nature has been. The cards definitely proved effective in New Guinea but this may have been due to the fact that they were mainly used by forestry units, to the personnel of which a certain degree of instruction had been given. No reports were received regarding the Philippine card sets, although from time to time the Division of Forest Products has received letters from individuals in U.S.A., the Philippines, and the Hawaian Islands who have referred to the key and have mentioned its value. Regarding the key for Malayan and East Indies timbers, again no very definite information is available. It did prove useful in Borneo where it was handled by officers of the Australian Army who had previously been trained on New Guinea timbers, but the war ended before it could be proved in Malaya.

However, the development of such card-sorting keys for field work under difficult conditions has been supported by Desch, who has recently (1946) referred to the war work of the Division of Forest Products in the field of timber identifications. Normand (1946) has similarly shown his interest and has made use of various features employed by us in order to develop a simple card-sorting key for French colonial timbers.

The fact that virtually untrained operators could, after short courses of instruction, carry out identifications with this type of key led to the consideration of the development of such a key for Australian commercial timbers and this project is now in hand. Preliminary indications are that by means of such ᵥ key the identification of these timbers will be within the reach of all foresters, forestry students, and timber users.

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The Variation of Tensile Strength and Modulus of Elasticity of Hoop Pine Veneer with the Direction of the Grain

*By R. S. T. Kingston, B.Sc., B.E.**

Summary.

Hoop pine veneer was tested in tension at various angles to the grain, the maximum tensile strength and the modulus of elasticity being determined.

Curves are included showing the variation of each property with the direction of the grain. These have been compared with a theoretical curve based on the assumption that wood is an orthotropic material in the case of modulus of elasticity and with a curve computed from the Hankinson formula in the case of maximum tensile strength.

For neither elastic modulus nor maximum tensile strength did the experimental points differ significantly from the computed curve.

1. Introduction

This investigation was designed to determine the effect of the angle between the direction of loading and the direction of the grain of veneer on its tensile strength and elastic modulus, and, using these data, to see how closely theoretical prediction of the tensile strength and elastic modulus in any given direction, based on measurements parallel and perpendicular to the grain, agreed with the experimental values.

Hoop pine veneer was used for the experiment. Thomas (8) found that there was no significant difference between the values for single veneer and two and three plies bonded together. Thus two or three ply material with the grain of all veneers in the same direction could have been used. One, two, and three veneers were all used in trials but, as it was found that single veneer could, with the exercise of a certain amount of care, be handled without damage, this was adopted.

2. Selection of Material, &c.

Material was selected from ten trees of hoop pine (*Araucaria cunninghamii* Ait.), one sheet of veneer being taken from each tree at random distances from the pith, all being 0·08 in. thick. The nominal test angles adopted were 0°, 5½°, 11¼°, 22½°, 45°, 67½°, and 90°. The sheets were marked in such a way that four test specimens were available from each sheet for each direction less than 30° to the grain and ten specimens from each sheet at each angle greater than 30° to the grain. It was found later that the curve was insufficiently determined around the 45° point, so further specimens were cut from the spare material at angles 35° and 55° to the grain, 10 per sheet in each direction, and also 1C at 45° as controls in case there was any slight difference betwe n widely separated parts of a sheet.

These specimens were all cut by hand with a specially sharpened knife in such a way as to minimize splitting of the veneer.

3. Method of Test

Half of the specimens from each sheet for each angle were tested with, and the remaining half without, an extensometer attached. A Gerrard extensometer was used, its weight being carefully balanced by means of a light spring so that the load on the specimen due to the extensometer was eliminated.

Fig. 1 shows the three types of specimens used. The specimens of the first type were held in the wedge grips of the machine in the usual way and were used for specimens with angles of 0°, 5½°, and 11¼° to the grain.

Those of the second type were held by pins and were used for the 22½°, 35°, 45°, and 55° specimens, the spring balance shown in Plate 1, Fig. 1, being used for the 35°, 45°, and 55° specimens, in which cases the testing machine was not sensitive enough to read the load with sufficient accuracy, and it was necessary to use a spring balance calibrated against dead weights.

Specimens of the third type were not fitted with end pads, as these were unnecessary on account of the low load. This type was used for the 67½° and 90° tests. In this case a special hand-operated
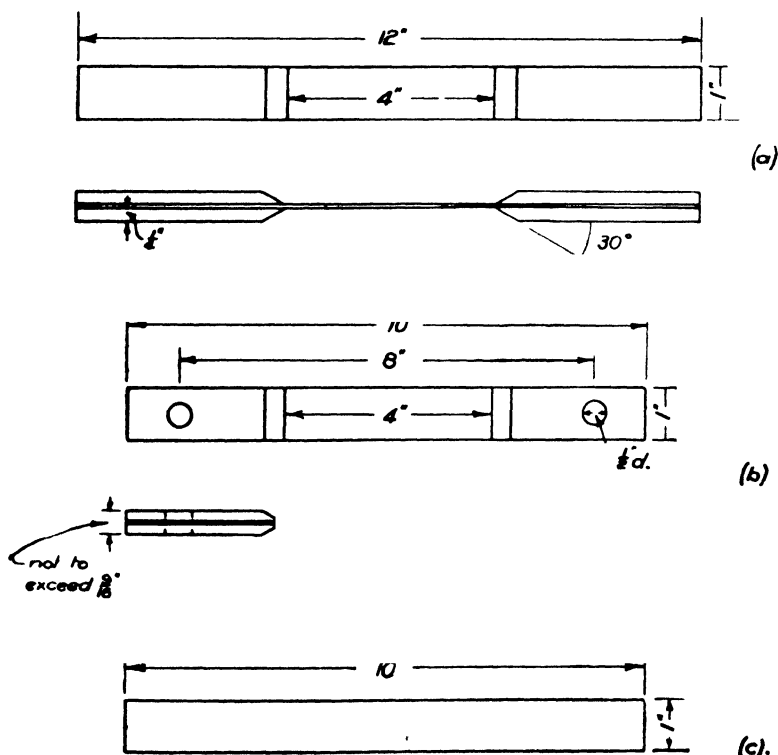
FIG. 1. (a) Test specimen with length at angles less than 22½° to the grain. (b) Test specimens with length from 22½°-55° to the grain. (c) Test specimens with length at 67½° and 90° to the grain.

machine was used, the load being determined by the deflection of a U-spring as measured by means of a dial gauge.

The material was conditioned in a room adjusted to a relative humidity which would bring the veneer to an equilibrium moisture content of 15 per cent. and was tested at a temperature of 21°C. ± 1°.

4. Results of Tests

The two groups of specimens tested at 45° to the grain showed no significant difference, so that all the results could be included without adjustment.

In Figs. 2 and 3, the maximum tensile strength and modulus of elasticity respectively have been plotted against the angle between the direction of loading and that of the grain. In each case the points plotted represent the average of all tests at each particular angle. In the case of tensile strength, results from specimens tested with an extensometer attached have been excluded on account of the possibility of damage caused by the gripping screws. In determining the average properties at each angle to the grain, specimens were culled in cases in which slight checks were present in the veneer.
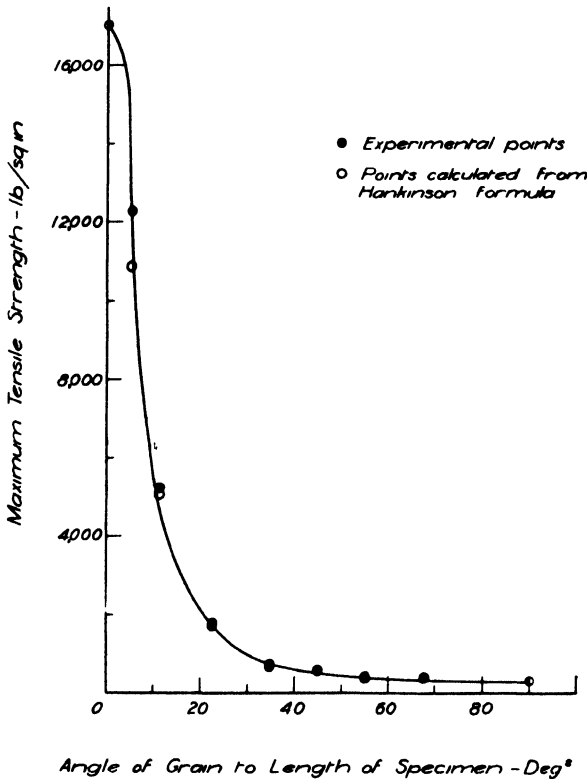
FIG. 2.—Variation in the tensile strength of hoop pine veneer with the angle between the direction of loading and the grain of the veneer.

It was especially necessary to do this carefully for tests with loading at large angles to the grain. A few very low values which may be due to variation in the tightness of peeling could not be definitely accounted for and were included in the results. Plate 1, Fig. 2, shows typical failures when the tension is parallel to the grain, and Plate 1, Fig. 3, with the tension at $5\frac{1}{2}°$ to the grain. At greater angles the failures were all very similar and one of each is illustrated in Plate 1, Fig. 4.

5. Discussion of Results

In addition to the experimental values, the maximum tensile strength and modulus of elasticity, as calculated from theoretical formulae, have been plotted in Figs. 2 and 3 against the angle between the direction of loading and that of the grain.
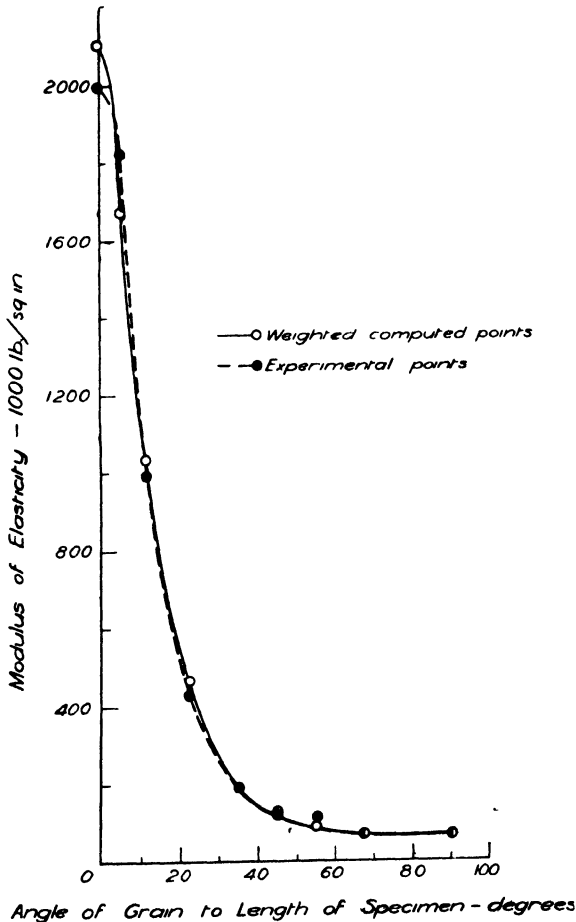
FIG. 3.—Variation in the elastic modulus of hoop pine veneer with the angle between the direction of loading and the grain of the veneer.

In the case of maximum tensile strength, the Hankinson formula has been used. This is as follows:—

$$N = \frac{PQ}{P \sin^2 \theta + Q \cos^2 \theta}$$

where N is the strength at an angle θ to the grain, P is the strength parallel to the grain, and Q is the strength perpendicular to the grain. This was put forward by Hankinson as an empirical formula and agrees very well with experimental results. This has been discussed by Langlands for wood in compression (2). He compared the Hankinson formula with others commonly used and showed that it gave, on the whole, better results than the others. He then compared it with his own experimental results and found a very close agreement. Since Langlands' paper was published, Norris (5) has shown that Hankinson's formula can be derived from Hencky's theory of failure (1, 4), by an approximate method. This places Hankinson's formula on a much more secure foundation and in view of these facts it was decided to use it here, in order to see how closely it agreed with the experimental results. It will be seen from Fig. 2 that the experimental and theoretical results do agree closely.

In the case of modulus of elasticity, assuming wood to be orthotropic, a value for the elastic modulus at any angle to the direction of the grain can be derived from the properties of the ellipsoids of stress and strain, and Hooke's law (3, 6). It is sufficient to consider the plane case only when dealing with thin sheets of peeled veneer. Roberts (7) has derived an expression which, together with Maxwell's theorem, gives the relationship used here. The result is, however, very simply derived from the basic equations for an orthotropic elastic body as given by Love (3).

Let XOY and $X'OY'$ be two sets of plane rectangular axes, the latter making an angle θ with the former. Let e_{xx}, e_{yy}, and $e_{x'x'}$, $e_{y'y'}$, be the normal strains parallel to the two sets of axes respectively whilst e_{xy}, $e_{x'y'}$, are the shear strains for these axes. Let X_x, Y_y, $X'_{x'}$, $Y'_{y'}$, X_y and $X'_{y'}$, be the stresses in corresponding directions. Let the undashed axes be the axes of elastic symmetry of the material, the x-axis being in the longitudinal direction and the y-axis in the tangential direction. Further, let E_x and E_y be the elastic moduli in the x and y directions respectively; σ_{xy} and σ_{yx} the Poisson's ratios respectively for strain in the y direction when stress is applied in the x direction and vice versa; and μ_{xy} the modulus of rigidity for shear in the xy plane. The stress-strain relations will then be as follows:—

$$
\left.
\begin{aligned}
e_{xx} &= \frac{1}{E_x} (X_x - \sigma_{xy} Y_y) \\
e_{yy} &= \frac{1}{E_y} (Y_y - \sigma_{yx} X_x) \\
e_{xy} &= \frac{1}{\mu_{xy}} X_y
\end{aligned}
\right\} \qquad \text{(I)}
$$

since. $\dfrac{\sigma_{xy}}{E_x} = \dfrac{\sigma_{yx}}{E_y}$ by Maxwell's theorem (7)

If now we express the stresses X_x, Y_y, and X_y in terms of X'_x, Y'_y, and X'_y the following relations hold (3).

$$X_x = X'_x \cos^2\theta + Y'_y \sin^2\theta - X'_y \sin2\theta$$
$$Y_y = X'_x \sin^2\theta + Y'_y \cos^2\theta + X'_y \sin2\theta$$
$$X_y = (X'_x - Y'_y) \sin\theta \cos\theta + X'_y \cos2\theta$$

(II)

The normal strain $e_{x'x'}$ in the direction of the OX' axis is given by:—

$$e_{x'x'} = e_{xx}\cos^2\theta + e_{yy}\sin\theta + e_{xy} \sin\theta \cos\theta \qquad (III)$$

Substituting the values of the strains as given by equation (I.) in equation (III.), inserting the values of X_x, Y_y and X_y as given by equation (II.), and considering the coefficient of X'_x the following is obtained:—

$$\frac{1}{E_x'} = \frac{\cos^4\theta}{E_x} + \frac{\sin^4\theta}{E_y} + \left\{ \frac{1}{\mu_{xy}} - \frac{2\sigma_{xy}}{E_x} \right\} \sin^2\theta\cos^2\theta .$$

Since the x-axis is in the longitudinal direction and the y-axis in the tangential, this can then be written:—

$$\frac{1}{E_\theta} = \frac{\cos^4\theta}{E_l} + \frac{\sin^4\theta}{E_t} + \left\{ \frac{1}{\mu_{lt}} - \frac{2\sigma_{lt}}{E_l} \right\} \sin^2\theta\cos^2\theta$$

where E_l and E_t are the elastic moduli of the veneer parallel to and perpendicular to the grain and E_θ is the elastic modulus at an angle of θ to the grain of the veneer. σ_{lt} is the Poisson's ratio for strain in the tangential direction when stress is applied in the longitudinal direction, and μ_{lt} is the modulus of rigidity for shear in the plane of the veneer.

Experimental values are not available for Poisson's ratios but as $\frac{2\sigma_{lt}}{E_l}$ comprises only a small percentage of the term containing it, the value of σ_{lt} for spruce, namely 0.54, can be used with little error. The theoretical curve of Fig. 3 has been fitted to the experimental data and the value for μ_{lt} for best fit determined. The most probable value appears to be 65,600 lb./sq. in. and using this value the experimental curve does not differ significantly from the fitted curve.

It was found that the means for large angles had a much greater error than those for small angles, despite the fact that more tests had been carried out for larger angles. In fitting the curve a transformation was therefore used. As the variances varied approximately as the square of the mean, the logarithms of the original values, rather than the values themselves, were analysed. The variance of the transformed values was found to be approximately constant. In general, the agreement appears to be reasonably close, the difference being quite small over the greater part of the range. Both in the case of maximum tensile strength and modulus of elasticity the ordinary theoretical formulae used, namely the Hankinson in the former case and the formulae for rotation of axes in material with orthotropic elastic properties in the latter case, give satisfactory

estimates of the values at various angles to the grain, provided the values parallel and perpendicular to the grain and the modulus of rigidity are known.

6. Acknowledgments

The author wishes to thank Mr. E. J. Williams who fitted curves to the experimental points and checked the statistical significance of the departure of the experimental points from the theoretical curves.

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Tests on Small Clear Specimens of North Queensland Kauri (*Agathis palmerstoni* F.v.M.)

By N. H. Kloot, B.Sc.*

Summary.

Mechanical tests have been carried out on five trees of North Queensland kauri. Analysis of the results in great detail was considered unwarranted in view of the small number of trees tested. However, the tests provide sufficient information for comparative purposes and in a comparison with two other species of similar density, viz., bunya pine (*Araucaria bidwilli* Hook.) and Sitka spruce (*Picea sitchensis* (Bong.) Carr), kauri has a lower strength/weight ratio for most of the important properties.

Although a mild form of compression wood was found in all five trees there did not appear any evidence that this affected the results.

1. Introduction

North Queensland kauri is the standard trade common name of the timber known botanically as *Agathis palmerstoni* F. v. M. and is the most important of the three kauris which grow in Queensland, the other two being *Agathis robusta* (C. Moore) F. M. Bail. and *Agathis microstachys* J. F. Bail. and C. T. White. As its name implies, it is a native of Northern Queensland where it is found chiefly on the slopes and in the highlands of the Atherton tableland. The tree is large, averaging 12 to 14 feet girth, breast high, and a total height of over 100 feet (6). An outstanding characteristic is the more or less cylindrical shape of the trunk, the taper being much less than in most species.

The timber ranges in colour from cream to pale reddish-brown. It is usually evenly grown and does not show definite growth rings and although softer and lighter is generally similar to the well-known New Zealand kauri (*Agathis australis* Salisb.).

An annual output of approximately 9 million super feet is absorbed in such fields as cabinet making, joinery, and internal sheeting and flooring of homes and railway carriages. The timber is also used for butter boxes, churns, and pats, for planking of light boats, for marine buoys and floats, and it has a high reputation in templet and pattern making.

At the request of the Queensland Forest Service a short series of tests was made on material from five trees of this species. As far as the author is aware, previous strength tests on kauri pine are scanty and of little practical value.

2. Material

Of the five trees collected for test, two came from the Kuranda district, two from the Little Mulgrave River district, and one from the Kairi district—all of which are within a radius of 50 miles from Cairns. One sawn flitch was selected from each tree, four being cut from middle logs and the fifth from a butt log. Details of the test logs are given in Table 1.

* An officer of the Division of Forest Products.

TABLE 1.—DETAILS OF LOGS TESTED.

Tree No.	Locality.	Annual Rainfall.	Approx. Elevation.	Slope.	Aspect (mag. bear.).	Soil, Subsoil, and Outcrops.	Type of Forest.	General Appearance of Tree.	Height to First Limb.	Height Overall.	Girth Breast Height.	Logs Selected for Test.
		in.	ft.						ft.	ft.		
1	T.R. 315, adjoining, Bor. 18. Parish of Smithfield. Situated about 40 chains from a large forest pocket. Kuranda district	90	1,350	1' in 15'	45° E.	Loose grey soil with clayey subsoil	Thick lawyer cane scrub	General taper, clean bole, and broken top. Had only one large limb when felled, the remaining top having been blown off in a cyclone or storm	64	84	11' 8"	Butt log, 12' long, stump 1' above ground
2	Part of T.R. 315, Bor. 281. Parish of Smithfield. Situated about 20 chains from a large forest pocket. Kuranda district	90	1,300	Approx. 1' in 1'	335° W.	Loose grey soil—clayey subsoil	Thick lawyer cane scrub	Standing almost perpendicular, good even taper, good top. Portion between first and second limbs, very knotty. Was on a very steep slope immediately above a gorge about 50' deep	64	96	11' 7"	Middle log, 12' long, stump 1' 3" at back and 6' at front above ground

TABLE 1.—DETAILS OF LOGS TESTED—*continued.*

Tree No.	Locality.	Annual Rainfall.	Approx. Elevation.	Slope.	Aspect (mag. bear)	Soil, Subsoil, and Outcrops	Type of Forest	General Appearance of Tree	Height to First Limb.	Height Overall	Girth Breast Height.	Logs Selected for Test.
		in.	ft.						ft.	ft.		
3	Blackwell Creek. R. 675. Parish of Grafton. Little Mulgrave River (Coastal) district	100	500	5°	360°	Grey stony soil	Poor type rain forest	Fine straight barrelled tree. Was growing near a small gully, in a slightly sheltered position	55	95	12' 7"	Middle log, 12' long, 15' above ground
4	Blackwell Creek. R. 675. Parish of Grafton. Little Mulgrave River (Coastal) district	100	500	10°	45°	Grey gravelly soil	Poor type rain forest	Fine straight barrelled tree which was dominating the surrounding forest	61	100	14'	Middle log, 12' long, 21' 6" above ground
5	R. 185. Danbulla. Karri (Tableland Area) district	75	2,500	..	..	Red soil	Dense lawyer vine	Good top—healthy tree Was standing in a valley	.	98	15' 2"	Middle log, 15' long

The sawn flitches, approximately 12 in. by 6 in., were resawn to yield 4 pairs of 2¼ in. by 2¼ in. tangentially-matched sticks, one stick in each pair being put aside for air-drying, the other for testing green. Specimens from both the dry and green sticks, after having been machined to 2 in. by 2 in., were subjected to the various tests according to the procedure, largely based on A.S.T.M. (1), B.S.I. (2), and British Air Ministry (3) specifications, adopted in this laboratory for the war-time testing of timbers for use in aircraft construction.

3. Correction for Moisture Content

Using an intersection point of 28 per cent. moisture content, the average relationship between strength and moisture content was calculated for each property using the U.S. Forest Products Laboratory exponential formula (7), and the estimated values at 12 per cent. moisture content obtained.*

All properties were corrected by this method with the exception of the two impact tests, toughness and Izod, for which the relationship between strength and moisture content is known to be considerably different from the exponential law for static properties. As yet, however, no tests have been done to determine correction factors for impact tests on this species.

4. Compression Wood

In common with other softwoods, kauri tends to develop compression wood and, in order to determine its severity in the trees tested, microscopic examination was made of specimens from every stick taken from each tree. A mild form of compression wood was found to be present in all the samples examined. It was not, however, sufficiently pronounced to be taken as definite compression wood. Where the latter did occur, it was found only in very narrow bands. These appeared more frequently in the two trees from the Little Mulgrave River district. Less was found in the trees from the Kuranda district, and the tree from the tableland area showed no evidence of definite compression wood.

5. Results of Tests

In Table 2 the average value for each property is given for the green and dry tests together with the appropriate statistical data. No figures are given for dry torsion as only one specimen was available for test, and in the green tests statistical data are not given for the same properties as only three specimens were tested.

In order to check the effect on the results of the compression wood in Tree 3 (the worst of the five trees in this respect) the strength values of the five trees were compared with one another; whilst the differences between trees were found to be significant, the effect was general and could not be attributed to one tree only. Consequently the results from all trees have been included in the species average and no result has been eliminated from any part of the analysis because of the presence of compression wood.

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* It will be noted that in Table 4 the results are quoted at 15 per cent. moisture content. This is due to the initial analysis of the results being made uniform with the procedure adopted for other species tested in the aircraft timber testing programme.

TABLE 2.—MECHANICAL AND PHYSICAL PROPERTIES OF SMALL CLEAR SPECIMENS OF NORTH QUEENSLAND KAURI.

Property.	Unit.	Moisture Condition	Species Mean	Standard Error of Species Mean	Standard Deviation of Tree Mean	Characteristics of Frequency Distribution				Approximate Correction for 1% m.c. for m.c.'s between 10% and 16%.
						Standard Deviation of Individual Results	Co-efficient of Variation per cent.	Observed Minimum	Observed Maximum	Number of Specimens	
Moisture content when green	%	..	47·7	..	..	..	..	..	..	..	..
Density	lb./cub ft	Green	37·1	1·24	2·76	2·95	8·0	32·2	42·5	20	..
		12%	29·0	0·96	2·14	2·23	7·7	25·3	32·3	19	0·17
Basic density (ratio of weight oven dry to volume green)	lb./cub ft		25·1	0·84	1·87	2·00	7·7	21·8	28·8	20	..
Shrinkage— Green to 12% moisture content—											
Radial	%	..	2·2	..			..	..	..	..	..
Tangential	%	..	3·4	..			..	..	..	..	..
Green to oven-dry—											
Radial	%	..	4·0	..			..	..	..	..	..
Tangential	%	..	5·6	..			..	..	..	..	..
Tension parallel to grain— 0·1% Proof stress	lb./sq. in.	Green	8,080	726	1,620	2,260	28·5	4,770	11,400	19	..
		12%	14,400	1,140	2,560	3,710	26·0	9,190	24,900	17	243
Maximum tensile strength	lb./sq. in.	Green	11,000	577	1,290	2,030	18·5	6,910	15,300	19	..
		12%	14,400	1,140	2,560	3,710	26·0	9,190	24,900	17	243
Modulus of elasticity	10^6 lb./sq. in.	Green	0·99	0·03	0·06	0·20	20·0	0·58	1·42	19	..
		12%	1·21	0·13	0·29	0·29	24·5	0·84	1·91	17	0·01

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TABLE 2.—MECHANICAL AND PHYSICAL PROPERTIES OF SMALL CLEAR SPECIMENS OF NORTH QUEENSLAND KAURI.—continue

Property	Unit	Moisture Condition	Specimen Mean	Standard Error of Specimen Mean	Standard Deviation of Tree Means	Standard Deviation of Individual Results	Coefficient of Variation per cent	Characteristics of Frequency Distribution			Approximate Corrections for 1% m.c. for m.c.'s between 10% and 16%
								Observed Minimum	Observed Maximum	Number of Specimens	
Compression parallel to grain (2" × 1" × 1" specimen)— Maximum crushing strength	lb/sq in	Green 12%	3,500 5,970	190 216	424 482	469 510	13·4 8·7	2,520 5,120	4,290 7,230	20 18	.. 176
Compression parallel to grain (8" × 2" × 2" specimen)— Stress at L.P.	lb/sq in	Green 12%	2,810 3,430	116 183	239 409	357 549	12·7 16·2	1,880 2,610	3,520 4,590	20 19	.. 39
Maximum crushing strength	lb/sq in	Green 12%	3,510 5,580	147 210	329 470	456 510	13·0 9·2	2,960 4,380	4,220 6,520	20 19	145
Modulus of elasticity	10⁶ lb/sq in	Green 12%	1·10 1·26	0·04 0·09	0·09 0·20	0·12 0·21	11·0 16·8	0·94 0·90	1·44 1·75	20 19	0·01
Compression perpendicular to grain (6" × 2" × 2" specimen)— Radial— Stress at L P	lb/sq in	Green 12%	481 709	31·6 36·2	70·6 80·9	93·2 86·1	19·4 12·2	327 586	650 838	12 10	.. 16
0·1% Proof stress	lb/sq in	Green 12%	556 933	39·4 37·1	88·1 82·9	88·0 86·8	15·8 9·3	475 796	702 1,080	12 10	.. 28

TABLE 2.—MECHANICAL AND PHYSICAL PROPERTIES OF SMALL CLEAR SPECIMENS OF NORTH QUEENSLAND KAURI.—continued.

Property.	Unit.	Moisture Condition.	Species Mean.	Standard Error of Species Mean.	Standard Deviation of Tree Means	Standard Deviation of Individual Results.	Co-efficient of Variation per cent	Observed Minimum.	Observed Maximum.	Number of Specimens.	Approximate Corrections for 1% m.c. for m.c.'s between 10% and 16%.
Compression perpendicular to grain (6″ x 2″ x 2″ specimen)—continued											
Tangential—											
Stress at L.P.	lb./sq. in.	Green	532	56.8	127	138	24.8	327	704	12	..
		12%	949	36.2	80.9	177	18.7	663	1,160	10	30
0.1% Proof stress	lb./sq. in.	Green	597	52.1	116	133	21.3	402	755	12	..
		12%	1,230	47.4	106	137	11.2	961	1,380	10	48
Compression perpendicular to grain (2″ x 2″ x 2″ specimen)—											
Radial—											
Stress at L.P.	lb./sq. in.	Green	284	33.7	75.3	85.4	30.0	175	512	16	..
		12%	..	..	..	..	..	..	..	..	..
0.1% Proof stress	lb./sq. in.	Green	342	38.2	85.5	88.4	25.7	219	532	16	..
		12%	..	..	..	..	..	..	..	..	..
Tangential—											
Stress at L.P.	lb./sq. in.	Green	298	22.5	50.2	68.1	22.7	151	402	16	..
		12%	..	..	..	..	..	..	..	..	..
0.1% Proof stress	lb./sq. in.	Green	372	30.1	67.2	76.6	20.4	202	503	16	..
		12%	..	..	..	..	..	..	..	..	..

TABLE 2.—MECHANICAL AND PHYSICAL PROPERTIES OF SMALL CLEAR SPECIMENS OF NORTH QUEENSLAND KAURI.—continued.

Property.	Unit.	Moisture Condition	Characteristics of Frequency Distribution.								Approximate Corrections for 1% m.c. for m.c.'s between 10% and 16%.
			Species Mean	Standard Error of Species Mean.	Standard Deviation of Tree Means.	Standard Deviation of Individual Results.	Co-efficient of Variation per cent	Observed Minimum.	Observed Maximum.	Number of Specimens.	
Static bending (4 pt. loading)— 0·1% Proof stress ..	lb./sq. in.	Green 12%	4,890 7,970	281 274	629 612	626 760	12·8 9·6	3,360 6,320	6,030 9,580	20 19	.. 245
Modulus of rupture .	lb./sq. in.	Green 12%	5,940 8,860	316 428	708 956	755 1,200	12·7 13·5	4,360 6,780	7,200 10,600	20 19	.. 204
Modulus of elasticity ..	10⁶ lb./sq. in	Green 12%	1·10 1·20	0·08 0·07	0·17 0·16	0·24 0·18	22·2 15·0	0·81 0·99	1·62 1·74	20 19	.. 0·01
Static bending (centre pt. loading)— Modulus of rupture ..	lb./sq. in.	Green 12%	6,710 9,210	302 437	675 978	888 1,100	13·1 11·8	4,940 7,350	8,240 11,000	12 11	.. 173
Modulus of elasticity ..	10⁶ lb./sq. in.	Green 12%	0·99 1·13	0·06 0·09	0·13 0·21	0·18 0·23	17·3 19·8	0·72 0·86	1·29 1·56	12 11	.. 0·01
Torsion— Stress at L.P. ..	lb./sq. in.	Green 12%	699 ..	..	..	..	..	604 ..	754 ..	3 ..	..
0·1% Proof stress ..	lb./sq in.	Green 12%	916 ..	..	..	..	..	869 ..	955 ..	3 ..	..
Maximum torsional strength ..	lb./sq. in	Green 12%	1,360 ..	..	..	..	..	1,260 ..	1,430 ..	3 ..	..
Modulus of rigidity ..	10³ lb./sq. in.	Green 12%	84·6 ..	..	..	..	..	77·4 ..	90·6 ..	3 ..	..

TABLE 2.—MECHANICAL AND PHYSICAL PROPERTIES OF SMALL CLEAR SPECIMENS OF NORTH QUEENSLAND KAURI.—continued.

Property.	Unit	Moisture Condition	Species Mean	Standard Error of Species Mean	Characteristic of Frequency Distribution						Approximate corrections for 1% m c for m c's between 10% and 16%
					Standard Deviation of Tree Means	Standard Deviation of Individual Results	Co-efficient of variation per cent	Observed Minimum	Observed Maximum	Number of Specimens	
Shear—											
Maximum strength—											
Plane of failure—Radial	lb./sq. in.	Green	769	49·9	112	115	14·8	570	1,040	15	· ·
		12%	1,130	47·0	105	130	11·5	901	1,350	10	24
Plane of failure—Tangential	lb./sq in	Green	853	48·0	107	108	12·5	670	1,020	15	· ·
		12%	1,110	46·1	103	111	10·0	885	1,240	10	16
Cleavage—											
Maximum strength—											
Plane of failure—Radial	lb./in.	Green	156	6·69	15·0	23·9	15·3	115	205	12	· ·
		12%	163	11·4	25·4	25·8	15·8	114	204	10	0
Plane of failure—Tangential	lb./in.	Green	178	5·16	11·5	31·9	17·7	143	251	11	· ·
		12%	374	11·1	24·9	34·0	9·1	327	449	10	15
Izod—											
Blow applied to radial face	ft. lb.	Green	3·9	1·14	2·56	2·49	63·7	1·3	9·4	20	· ·
		*	1·8	0·08	0·19	0·35	19·4	1·2	2·5	19	· ·
Blow applied to tangential face	ft. lb	Green	4·2	1·18	2·65	2·71	64·7	1·2	11·5	20	· ·
		*	1·7	0·18	0·41	0·43	25·3	1·1	2·9	19	· ·
Toughness—											
Blow applied to radial face	in lb.	Green	99·3	13·7	30·7	33·7	34·0	50·2	160	20	· ·
		*	50·4	3·40	7·61	12·8	25·4	19·4	71·0	19	· ·
Blow applied to tangential face	in lb	Green	105	10·7	23·9	35·6	33·8	67·4	172	20	· ·
		*	50·9	5·50	12·3	14·6	28·7	26·0	72·3	19	· ·
Hardness—											
Tangential	lb.	Green	526	35·4	79·2	85·8	16·4	370	675	20	· ·
		12%	516	34·8	77·1	84·6	16·4	360	625	19	0

* Average moisture content approximately 16%.

In Plate 2, Figs. 1, 2, and 3 illustrate typical failures of dry kauri pine in centre-point bending, four-point bending, compression parallel to the grain, Izod, and toughness. The pronounced brittle failures in both static and impact bending tests are reflected in the very low impact values.

As the number of specimens tested to determine any particular property was small, it was considered that the preparation of frequency polygons would not serve any useful purpose and might prove misleading.

6. Correlations Between Properties

In attempting to select material with certain minimum strength requirements, it is of advantage to fix on an easily determined property such as density and knowing the correlation between this and the desired properties, to select the timber within a given range or above a given minimum value of this property. This is usually fairly difficult with timber owing to the low correlation obtained between most pairs of properties.

In Table 3 a list is given of those correlation coefficients which proved to be statistically significant. Both dry and green results were analysed together and, where the difference between moisture contents was shown to be not significant, the total correlation and a single regression for both sets of results are given. Where the difference in correlation coefficients for dry and green tests proved to be significant, regressions for both conditions are given and the correlation coefficient with moisture condition eliminated is also shown in place of a total correlation coefficient. Most of the numerically large coefficients are of little practical importance, e.g. radial Izod v. tangential Izod, whereas those which might be of some practical importance are generally too low to be of much assistance in selection. An example of the latter is the correlation between density and maximum crushing strength, which is shown as $0 \cdot 748$. This means that only $0 \cdot 748^2 \times 100$ or 56 per cent. of the variation in crushing strength is attributable to variation in density.

In Figs. 1 to 6, the results from some of the tests are plotted against density and, with the exception of tensile strength against density where the correlation was not significant, regression curves have been included. It must be emphasized that the total number of test values is relatively small and this analysis can only serve as a guide until a much wider range of material has been tested to obtain a more accurate estimate of the properties and the relationships between properties of this species.

7. Comparison with Other Species

In Table 4 a comparison is made between kauri pine and two other species of similar density, viz. bunya pine and Sitka spruce, the data for which were obtained from an unpublished report on the properties of bunya pine. On the whole, kauri pine does not compare favourably with either of the other two species, particularly in tensile strength, modulus of elasticity, and impact strength. Except in compression

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RELATION BETWEEN MECHANICAL PROPERTIES AND DENSITY

FIG 1
Maximum Crushing Strength (Large Prism) lbs/in²
Y=471+767 Dry
Y=471+489 Green
Density—lb/cub Ft (X)

FIG 2
Maximum Crushing Strength (Small Prism) lbs/in²
Y=1821+25 Dry
Y=1821+1010 Green
Density—lb/cub Ft (X)
KEY Green Spec., Dry Spec me

FIG 3
Modulus of Rupture—lbs/in² (Y)
Y=326+2290 Green
Y=326+650 Dry
Density—lb/cub Ft (X)

FIG 4
Maximum Tensile Strength—lbs/in² (Y)
No Significant Regression
Density—lb/cub Ft (X)

FIG 5
Hardness—lb (Y)
Y=3.6x 85 Dry
Y=3.6 x 243 Green
Density—lb/cub Ft (X)

FIG 6
Radial Shear—lb/sq in (Y)
Y=56.9 x 628
Density—lb/cub Ft (Y)

TABLE 3.—NORTH QUEENSLAND KAURI—REGRESSIONS AND CORRELATIONS.

Independent Variable (x).		Dependent Variable (y).		Significance of Difference between Moisture Conditions.	Linear Regression of y on x.	Correlation Coefficients.		
Property.	Unit.	Property	Unit.			Total.	Within Trees.	Total (Moisture Condition Eliminated)
Maximum crushing strength (small prism)	lb./sq. in.	Hardness	lb.	**	Y (green) = ·0138x + 478 Y (dry) = ·0138x + 435	..	·562**	·804**
		Maximum crushing strength (large prism)	lb./sq. in.	**	Y (green) = ·691x + 1,040 Y (dry) = ·691x + 1,420	..	·479**	·980**
		Radial toughness	in. lb.	**	Y (green) = ·0246x + 13·4 Y (dry) = ·0246x + 81·9	..	·422*	·512**
		Modulus of rupture	lb./sq. in.	‡‡	Y = 1·24x + 1,610	·875**	·574**	..
		Modulus of elasticity (static bending)	10³ lb./sq. in	‡‡	Y = ·0813x + 782	·408*	·384*	..
Density	lb./cub. ft.	Maximum crushing strength (small prism)	lb./sq. in.	**	Y (green) = 182x + 1,010 Y (dry) = 182x + 24·8		·613**	·249‡
		Hardness	lb.	**	Y (green) = 31·6x − 243 Y (dry) = 31·6x − 415	..	·662**	·964**
		Maximum crushing strength (large prism)	lb./sq. in.	**	Y (green) = 147x − 189 Y (dry) = 147x − 767	..	·312‡	·748**
		Radial toughness	in. lb.	**	Y (green) = 5·35x − 31·1 Y (dry) = 5·35x − 109·8	..	·596**	·506**

** Significant at the 1 per cent level of probability.
* Significant at the 5 per cent level of probability
‡ Not significant

TABLE 3.—NORTH QUEENSLAND KAURI—REGRESSIONS AND CORRELATIONS.—*continued.*

Independent Variable (x)		Dependent Variable (y)		Significance of Difference between Moisture Conditions	Linear Regression of y on x	Correlation Coefficients		
Property	Unit	Property	Unit			Total	Within Trees	Total (Moisture Condition Eliminated)
Density	lb/cub ft	Radial shear	lb/sq in	++	Y = 56·4x − 628	·914**	·687**	
		Modulus of rupture	lb/sq in	**	Y (green) = 326x − 2,290; Y (dry) = 326x − 1,540		·415*	·076‡
Radial Izod	ft lb	Tangential Izod	ft lb	++	Y = 1·08x − ·132	·965**	·746**	
		Radial toughness	in lb	**	Y (green) = 10·2x + 59·6; Y (dry) = 10·2x + 32·5		·245‡	·690**
Radial toughness	in lb	Tangential toughness	in lb	++	Y = 1·00x + 2·87	·909**	·744**	
		Maximum tensile strength	lb/sq in	*	Y (green) = 48·3x + 6,210; Y (dry) = 48·3x + 11,100		·402*	·407*
		Modulus of rupture	lb/sq in	**	Y (green) = 13·2x + 4,640; Y (dry) = 13·2x + 7,560		·240‡	·387*
Maximum tensile strength	lb/sq in	Modulus of rupture	lb/sq in	**	Y (green) = ·175x + 4,010; Y (dry) = ·175x − 5,760		·424*	·513**

** Significant at the 1 per cent level of probability
* Significant at the 5 per cent level of probability
‡ Not significant.

TABLE 4.—COMPARISON OF AVERAGE PROPERTIES OF SMALL CLEAR SPECIMENS (UNSELECTED) OF NORTH QUEENSLAND KAURI, BUNYA PINE, AND SITKA SPRUCE AT 15 PER CENT. MOISTURE CONTENT.

Property	Unit	Species Average			Average/G *			Average/G$^{3/2}$			Average/G^3		
		Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce
Density	lb cub ft	29·5	29·1	28							·	·	·
Air dry specific gravity at 15% M C (G)		0·472	0·466	0·448							·	·	·
Shrinkage to 12% M C— Radial	per cent	2·2	2·0	2·5									
Tangential	per cent	3·4	4·0	4·0							·		·
Tension parallel to grain— Ultimate strength	lb/sq in	13,700	16,700	18,000	29,100	35,800	40,900						
Compression parallel to grain— Ultimate strength (2" x 1" x 1' specimen)	lb/sq in	5,420	6,200	4,900	11,500	13,300	11,100						
Modulus of elasticity	10⁶ lb/sq in	1·23	1·84	1·80 (est.)	2·61	3·94	4·10				5·53	8·44	9·30
Compression perpendicular to grain (6" x 2" x 2" specimen), load applied to radial face— Stress at L P	lb/sq in	660	600	600	1,400	1,300	1,360						
Static bending (4 pt loading)— Modulus of rupture	lb/sq in	8,220	9,400	8,300 (est.)	17,800	20,200	19,000	24,000	29,500	28,000			

* G = Air dry specific gravity at 15 per cent moisture content

TABLE 4.—COMPARISON OF AVERAGE PROPERTIES OF SMALL CLEAR SPECIMENS (UNSELECTED) OF NORTH QUEENSLAND KAURI, BUNYA PINE, AND SITKA SPRUCE AT 15 PER CENT MOISTURE CONTENT—continued.

Property	Unit	Species Average			Average/G, *			Average/$G^{2/3}$			Average/G^2		
		Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce	Kauri	Bunya Pine	Sitka Spruce
Torsion—													
Ultimate torsional shear strength	lb/sq in		1,790	1,360		3,830	3,090		5,600	4,630		450	520
Modulus of rigidity	10³ lb/sq in		98·5	100		210	230						
Shear parallel to grain—													
Ultimate strength (average radial and tangential)	lb/sq in	1,060	1,200	1,020	2,250	2,600	2,320						
Cleavage—													
Ultimate strength (average radial and tangential)	lb/in	245	190	210	520	410	480						
Izod—													
Blow applied to radial face	ft lb	1·8	4·0	5·5	3·8	8·6	12·5						
Toughness—													
Blow applied to radial face	in lb	50	64	83	107	140	190						
Hardness—													
Tangential face	lb	520	500	480	1,100	1,070	1,090						

* G Air dry specific gravity at 15 per cent moisture content

parallel and perpendicular to the grain and one or two minor properties, kauri is inferior to both bunya pine and spruce and in either of these two species a more efficient solid Euler strut (strength/density^2) or a more efficient solid spar (strength/density$^{3/2}$) may be constructed.

8. Properties Other Than Strength

Experiments on the steam bending qualities of this species (5) show it to be inferior to such species as celery-top pine (*Phyllocladus rhomboidalis* L.C. Rich.), radiata pine (*Pinus radiata* D. Don) and Corsican pine (*Pinus laricio* Poir.). Like hoop pine, kauri has little resistance to decay.

In general, kauri has many desirable qualities as evidenced by the wide range of uses to which this species is put. It is characteristically clear and straight-grained and little difficulty is encountered in its seasoning. It machines and glues well and its almost complete lack of odour and " taint " makes it an ideal medium for food containers such as butter boxes. Data given by Greenhill (4) indicate that kauri has a low shrinkage of the order of 0·2 per cent. per 1 per cent. moisture content change in both the radial and tangential directions.

9. References

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The Nature of Plastic Deformation in Wood at Elevated Temperatures

By H. G. Higgins, B.Sc. (Hons.), and Florence V. Griffin.**

Summary.

1. Elastic after-effects were observed in hoop pine plywood panels which had been oven-dried and subjected for five minutes to pressures capable of causing residual deformation at elevated temperatures.

2. For the major residual deformations observed, elastic after-effect comprised about half of the total residual deformation at a pressing temperature of 120°C., a proportion which declined to one-seventh at 200°C.

3. The influence of elastic after-effect was such as not to alter radically the effect of temperature upon yield-value.

4. Microscopic studies showed that the radial pressure required to produce fracture of the cell wall declined with increase in temperature, the effect being linear over the range examined, with a regression coefficient of —6·75 lb./sq. in. per degree C.

5. From a consideration of temperature and pressure effects, three main zones of deformation could be defined: those of elasticity, flow, and fracture. The zone of flow was divisible further into areas of plasto-elasticity and visco-elasticity.

6. It appeared probable that the location of flow in the wood tissue was the cell wall as well as the middle lamella zone.

7. Deformation in the zone of flow appeared to involve re-arrangement on a sub-microscopic scale, insofar as gross fracture of the cell wall was absent, and minute compression failures were not significantly more abundant than in the zone of elasticity.

8. Rheological studies may contribute to the knowledge of the structure of the cell wall. Short loading times and elevated temperatures should be used in conjunction with sustained loading at ordinary temperatures.

1. Introduction

In a previous paper (1) an experiment, which was designed to investigate the effect of temperature upon the immediate residual deformation of plywood panels pressed for a fixed time under varying loads, was described. It was found that, for oven-dry material, the load at which appreciable residual deformation took place declined rapidly with rising temperature from 120° to 160°C., and was small over 160°C. The virtual disappearance of a yield value near this temperature appeared to represent a fundamental change in plastic behaviour.

Two major problems were suggested by the results of this investigation: firstly, what part of the immediate residual deformation can be attributed to elastic rather than plastic effects? and secondly, what is the physical nature of so-called plastic deformation in a material of such complexity as wood?

The answer to the first question would involve the measurement of elastic recovery in the original specimens after the lapse of a considerable period. This would permit due allowance to be made

for the elastic after-effect in defining the dependence of yield value upon temperature, and the results could be compared with those obtained by measuring residual deformation immediately after pressing.

The second problem sought confirmation or otherwise of the implied hypothesis (1) that each residual deformation was due, in part at least, to plastic behaviour of the material of the wood, and was not simply the result of crushing, i.e., fracture, of the cells. Some evidence was provided in support of this view in that temperature critically controls the mode of deformation, an apparently plastic or quasi-plastic behaviour being superseded by viscous or quasi-viscous flow above about 160°C., and it was remarked that this temperature accorded with the softening point of some dry extracted lignins. It was the aim of the present investigation to obtain further evidence directly of the nature of plastic deformation by microscopic examination of sections of the specimens.

2. Materials

The material used for this experiment was identical with that used previously (1), and consisted of plywood specimens 4 in. by 4 in., made up from three veneers, of nominal thickness 1/16 in., of rotary-cut hoop pine (*Araucaria cunninghamii* Ait.), bonded together at 50 lb./sq. in. and 145°C. for eight minutes with a phenol-formaldehyde film adhesive. All the specimens had been cut from the same plywood panel, and the veneer consisted of closely matched material from the same log. The specimens had been subjected to pressures ranging from 200 to 1,200 lb./sq. in. over a temperature range of from 120° to 200°C. Each specimen had been oven-dried and then pre-heated for two minutes before the application of the load, which had been maintained for five minutes in every case. Residual deformation had been measured by obtaining the differences in thickness immediately before insertion in and immediately after removal from the press.

3. Determination of Elastic After-effect

After the lapse of periods of 55 and 100 days from pressing, the thickness of each specimen was re-measured, using a micrometer gauge as before, each determination being the mean of eight measurements—one in each corner and one near the centre of each side. The specimens were oven-dried before measuring in order to reproduce the original moisture content and to eliminate dimensional changes due to the absorption of moisture since pressing. However, most of the recovery must have taken place at a moisture content in equilibrium with laboratory conditions.

4. Influence of Elastic After-effect on Yield Line

The elastic after-effects are shown in Table 1, and it can be seen that the major part of the delayed recovery occurs in the first period of 55 days after pressing, indicating that the total effect converges to a finite limit, which will be a small multiple of the recovery observed in the first period.

TABLE 1.—RESIDUAL DEFORMATION AND ELASTIC AFTER-EFFECT.

	Pressure (lb /sq in)	Temperature (° C)														
—		120			140			160			180			200.		
		a	*b*	*c*	*a*	*b*	*c*	*a*	*b*	*c*	*a*	*b*	*c*	*a*	*b*	*c*
Total Residual	200	5	8		3	2		5	5	*	8	10	6	3	8	2
Deformations	400	2	3		5	3		11	5	*	22	17	13	18	21	15
(In × 10⁻³)	600	2	0		4	2		23	15	*	36	27	21	81	70	68
Compression	800	5	−6		11	7	6	19	14	7	56	45	36	235	209	204
Positive	1,000	2	2		33	25	21	60	38	30	232	177	168	505	449	447
	1,200	43	21	24	89	60	55	260	200	187	414	320	304	639	554	559
Total Elastic	200	0	−3		0	1		0	0		0	−2	2	0	−5	1
After-effect	400	0	−1		0	2		0	6	*	0	5	9	0	−3	3
(In. × 10⁻³)	600	0	2		0	2		0	8	*	0	9	15	0	11	13
Recovery	800	0	11		0	4	5	0	5	12	0	11	20	0	26	31
Positive	1,000	0	0		0	8	12	0	22	30	0	55	64	0	56	58
	1,200	0	22	19	0	29	34	0	60	73	0	94	110	0	85	80

a = Immediately after pressing   *b* = 55 days after pressing   *c* = 100 days after pressing.   * Specimens delaminated

If the residual deformations after 55 days, for example, are plotted against the pressure, for each temperature, a family of curves is obtained (see Fig. 1), which is similar to that obtained for the
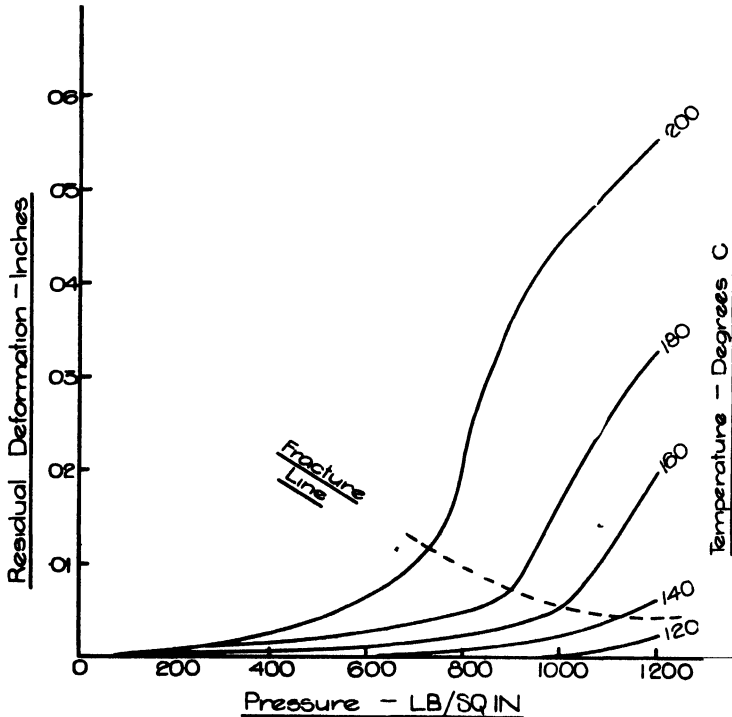
FIG. 1.—Residual deformation 55 days after pressing.

immediate residual deformations (1). From these new curves a new yield value versus temperature curve ("yield line") can be drawn, the yield values being taken, as before, as the pressures corresponding to the points at which the smoothed curves in Fig. 1 approach the load axis (or, in practice, intersect the + ·0005 in. abscissa). The new yield line (Fig. 5—lower line) is similar to that obtained using immediate residual deformations.

The dependence of elastic after-effect upon both pressure and temperature is defined, in Fig. 2, by contours on the surface representing the empirical function which expresses that dependence. The
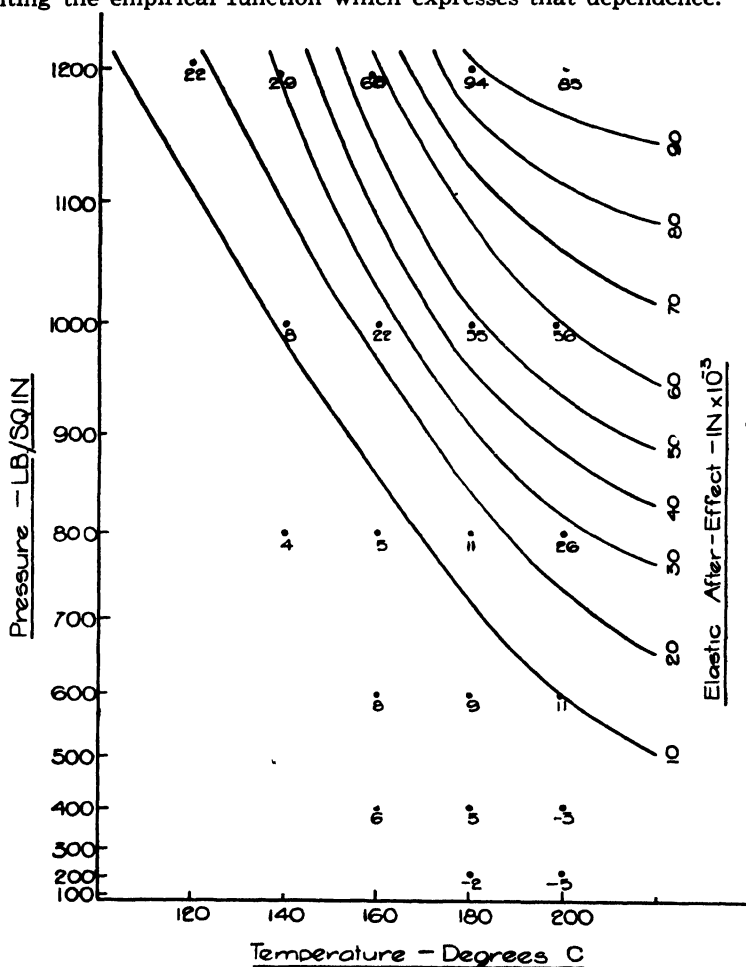
FIG. 2.—Elastic after-effect 55 days from pressing.

contours have been drawn from the elastic after-effects at 55 days, and, to facilitate interpretation, the square of the pressure has been plotted along the vertical axis. Since the temperature and moisture content of the specimens during the recovery period differed from those during pressing, the diagram can be considered as of only practical significance.

5. Effect of Temperature on Elastic After-effect

Although quite valid practically, Fig. 2 gives a somewhat false theoretical conception of the dependence of elastic after-effect upon pressing temperature, since the apparent increase of one with the other at constant pressure is due to the great increase in plasticity with rising temperature. This may be shown by the simple expedient of determining what proportion of the total initial residual deformation is due to elastic after-effect at each temperature. Table 2 shows this proportion expressed as a percentage and, in order to eliminate experimental inconsistencies for small measurements, the ratio has been computed only for elastic after-effects greater than 0·001 in., and at pressures and temperatures greater than the yield value.

TABLE 2.—RATIO OF ELASTIC AFTER-EFFECT TO TOTAL INITIAL RESIDUAL DEFORMATION (PER CENT.).

Pressure (lb./sq. in.)	Temperature (°C)									
	120		140		160		180		200	
	b	c	b	c	b	c	b	c	b	c
600	.	..	..		..	.	..		14	16
800	.	..	..	.		..	30	34	11	13
1,000		.	.		37	50	24	28	11	11
1,200	51	44	33	38	23	29	23	27	13	12
Time Means	51	44	33	38	30	40	26	30	12	13
Temp. Means	48		36		35		28		13	

b = 55 days after pressing   c = 100 days after pressing

The correlation between temperature and the ratio of elastic after-effect to total initial residual deformation is expressed in Fig. 3, which is plotted from the figures in Table 2. It now becomes apparent that elastic after-effect decreases in importance with rising temperature, rather than increasing as suggested by Fig. 2. It is probable that delayed elastic recovery was inhibited to some extent by the cooling of the specimens after pressing.

6. The Relationship between Types of Deformation and Organization of the Cell Wall

The continued existence of elastic after-effects with pressing temperatures above that at which the yield value becomes small, may shed light on the general nature of the deformation above this critical temperature (160°-170°C.). This elasticity immediately excludes, as might, of course, be expected for wood, even at high temperatures, the possibility of Newtonian or true viscous flow, and it indicates further that the behaviour at such temperatures is of the visco-elastic type. The terms used here are in accordance with the classification of strains proposed by the British Rheologists' Club (4) and discussed by Scott Blair (6, p. 43).

Similarly, below the critical temperature, since elastic after-effects cannot occur in any plasto-inelastic system, of which the Bingham solid is a special case, the deformation must therefore be of the plasto-elastic type, as it is in wood at ordinary temperatures.
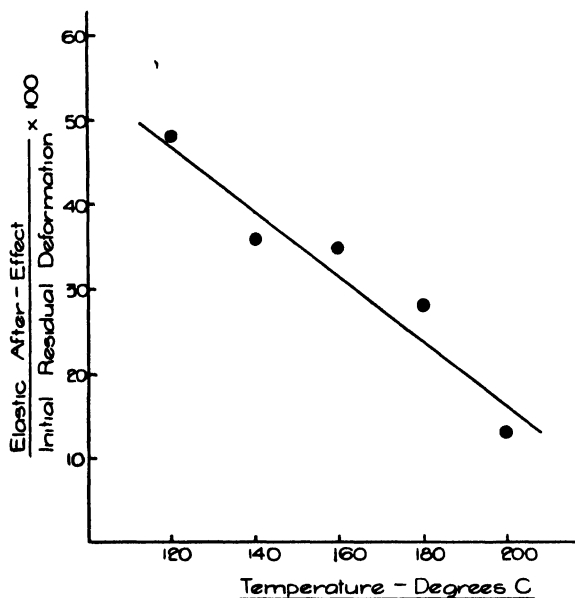
FIG. 3.—Effect of temperature on relative elastic after-effect.

Although by no means conclusive, it seems reasonable to suggest that visco-elastic or plasto-elastic flow would be much more likely to occur in a system consisting of two (or more) discrete materials, perhaps quite intimately linked, but nevertheless maintaining to some degree their individual response to deforming stresses. If definite evidence can be adduced along these lines, it may be possible to draw important conclusions concerning the organization of lignin and of carbohydrates in the plant cell wall. It will be necessary to demonstrate that flow actually takes place in the cell wall, and not merely in the middle lamella zone, a postulate which will be defended later.

Houwink (2, p. 250) points out, moreover, that correlation between the orientation and length of the micelles and the shape of the deformation curves has been established in the case of some cellulosic materials. Van Iterson (9) for instance studied the deformation curves for cellophane in different directions, and found distinct differences which he ascribed to orientation of the micelles. The method could perhaps be applied analytically for natural materials.

7. Microscopic Examination of the Cell Wall

Two sections of each plywood specimen were cut and mounted on slides in Canada balsam. One section of each pair showed a cross-section of the centre veneer and a radial section of the two

face veneers, while the other section, cut at right angles to the first, showed the centre veneer in radial section and the faces in cross-section.

Examination of the sections was made under ordinary light, and in random order so as to eliminate any possible personal bias, with a view to determining in which specimens crushing or fracture had taken place. Radial sections yielded little or no information in this regard, but the cross-sections were clearly divisible into two groups. One showed no evidence of crushing, in the sense of fracture of the cell wall, although in some specimens deformation of the tracheids had apparently taken place in a manner which could have been attributed to a flow phenomenon. The other group showed definite bands in which complete fracture of the cell wall had taken place.

Plate 3 (*a*) shows a photo-micrograph of a specimen without fracture. Plate 3 (*b*) and (*c*), by way of contrast, displays cell wall fracture in different degrees of advancement. Incipient in 3 (*b*), it is very much advanced in 3 (*c*), where the orientation of the fracture zones into parallel bands is no longer evident.

It can be seen, by examination of the less fractured specimens, that this orientation is due to fracture occurring parallel to the growth rings, and, as would be expected, it is in the large, thin-walled cells of the early wood that fracture takes place initially. This is shown in Plate 3 (*b*).

The specimens were examined further in polarized light, with the object of determining the incidence of slip planes and minute compression failures. Slip planes were observed in all sections, and minute compression failures only in those cut from specimens which had been pressed under the following sets of conditions:—

Pressure (lb./sq. in.)	200	400	400	600	800	1,000	1,000	1,200	1,200	1,200
Temperature (C.)	160	120	180	140	200	140	200	120	140	160

8. Effect of Pressure and Temperature on Cell Wall Fracture

Table 3 lists the temperatures and pressures to which specimens of the two groups were subjected. Except where otherwise indicated, the cell wall fracture occurred in none or all of the veneers of the plywood. The values of the pressure at which fracture occurred (see column headed "Interpolated from Observations") represent the pressure at which fracture would be expected to occur in 1·5 veneers, i.e., in half the total number. When these values of "fracture pressure" were statistically examined in relation to temperature, a linear regression was established with a coefficient of —6·75 lb./sq. in. per degree C., which gave the estimated values shown in the last column of Table 3. The regression was computed by Mr. E. J. Williams of the Section of Mathematical Statistics, C.S.I.R.

These results are shown graphically in Fig. 4 in which the abrupt and approximately linear nature of the boundary between the zones of fracture and of non-fracture on the pressure-temperature diagram

is clearly seen.   It is of interest to note that if the fracture pressures for each temperature are plotted on the pressure versus residual deformation curves for any period after pressing (e.g., 55 days, as in Fig. 1) then the points obtained are those at which the slope of the isothermals rapidly increases, thus providing the physical explanation for that phenomenon.   These points are joined by the broken line in Fig. 1.

TABLE 3.—VARIATION OF " FRACTURE PRESSURE " WITH TEMPERATURE.

Temp (° C)	Pressures Applied to Specimens Showing no Fracture (lb /sq in)	Pressures Applied to Specimens Showing Fracture (lb /sq in)	Pressure at which Fracture Occurs (lb /sq in).	
			Interpolated from Observations	From Linear Regression
120	200, 400, 600, 800, 1,000, 1,200 ..	. .. ..	1,200	1,275
140	200, 400, 600, 800, 1,000* . ..	1,200† . ..	1,100	1,140
160	200, 400, 600, 800, 1,000*	1,200	1,050	1,005
180	200, 400, 600, 800 ..	1,000, 1,200	900	870
200	200, 400, 600 ..	800, 1,000, 1,200 ..	700	735

* Fracture in one veneer   † Fracture in two veneers.
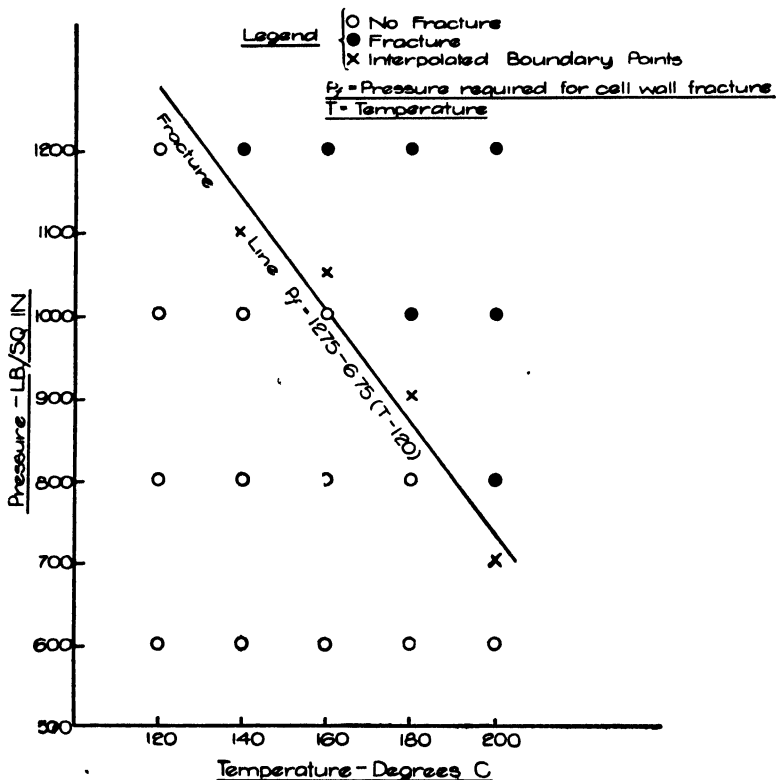
FIG. 4.—Effect of temperature on strength of cell wall.

9. Principal Zones of Deformation

The effect of temperature upon both yield value and "fracture pressure" is shown in Fig. 5, the yield line being arbitrarily selected as that corresponding to conditions 55 days after pressing, and the fracture line being taken directly from Fig. 4.

Three major zones of deformation are now defined. The lower zone is that of elastic deformation: nothing can be said from the present data as to whether further boundaries can be drawn within the zone, e.g., between Hookean and non-Hookean deformation. However, it may be noted that the proportional limit can never exceed the yield value and can only equal it if all the elastic deformation follows Hooke's Law—a rather improbable condition, perhaps, in dry wood at any temperature. The upper zone is that of fracture of the cell wall, and as such is practically beyond rheological study, although, in compression, complex plastic and elastic effects will continue to operate with increasing stress, even as failure proceeds. Our main interest in this zone at present, however, is in its lower boundary, and in particular in the large distance by which that line is removed from the yield line, and even from the position the yield line would be expected to occupy after the final appreciable dissipation of elastic after-effect.

Zone of
Cell Wall
Fracture

Fracture Line

Zone of Flow

Plasto-
Elasticity

Yield Line

Zone of
Elastic
Deformation

Visco-
Elasticity

Pressure LB/SQ IN

1200 1000 800 600 400 200

Temperature – Degrees C

120 140 160 180 200
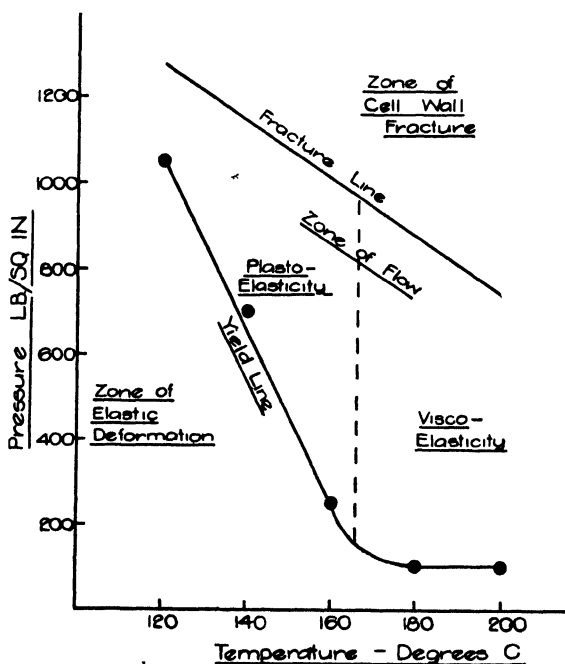
FIG. 5.—Zones of deformation.

It is in the middle zone of Fig. 5, then, between the fracture line and the yield line that plastic deformation is of major importance. A further sub-division of this zone can be made at the critical temperature into plasto-elastic and visco-elastic deformation. It seems fairly

reasonable to assume that this boundary is an isothermal, since the type of flow should not depend on stress, within such narrow limits, once the yield value is exceeded.

The question of the location of flow deformation within the woody tissue is worthy of passing consideration, although the evidence is somewhat limited. From simple geometrical considerations, it would appear that the very fact that permanent external volumetric compression takes place under stress, without fracture of the cell wall, indicates that the deformation cannot be solely in the middle lamella zone. It seems justifiable to conclude that flow must also take place in the material of the cell wall, which comprises the main bulk of the tissue. (Support for this view is perhaps lent by the recent conclusion of Wardrop and Dadswell (10) that intercellular adhesion in *delignified* tissue is to be attributed mainly to direct mechanical adhesion between the cells, and that the previous conception of a dilute alkali soluble, non-lignin, non-polyuronide bonding material must be rejected.)

Reverting to Fig. 5, one practical application of the division of the pressure-temperature diagram into zones of deformation may lie in defining conditions for the manufacture of densified wood products having optimum strength/weight ratios. For a particular species, time of pressing, and moisture content, it would be necessary to select temperature and pressure so as to exceed the yield line in order to obtain permanent densification. On the other hand it seems very probable that strength properties will not continue to increase much more rapidly than density if the fracture line is exceeded. Best results could possibly be obtained at temperatures and pressures just below the fracture line.

10. General Discussion.

Some diffidence has been felt by one of the present writers in a previous paper (1) in using the term " yield value " to connote the compressive stress at which appreciable permanent deformation takes place, a meaning which has been retained in these discussions. Various definitions of the " yield value " of some materials, usually based on the tensile stress required for an arbitrary small permanent extension of a rod of fixed length, have been proposed. There seems to be no general agreement among rheologists as to what is meant by the term (6, p. 184) and indeed the acceptance or otherwise of the concept of " yield value " forms the basis of the difference between the analytical and integralist schools in rheology. So the term " yield value " is used here in its widest sense with full cognizance of the necessarily arbitrary conditions imposed by the present tests: namely, the moisture content of the wood, the time and rate of loading, the species, &c. However, the qualitative picture may be of some use.

The short loading time used need not be considered as incompatible with generally applicable conclusions on the plasticity of wood. Insofar as elevated temperatures have an effect similar to long-time loading in increasing residual deformation, the study of wood at these temperatures can provide data for correlation with the study of creep at ordinary temperatures, and of course these data can be much more quickly obtained. Tests conducted at ordinary temperatures

and with long-time loading are of vital importance for direct application to problems in constructional engineering, as Kingston (3) points out, tests at elevated temperatures with short loading time are more applicable in practice to the manufacture of derived wood products, e g , plywood and densified wood, and plastics containing wood flour For clarification of the theory of flow in wood, both lines of attack are necessary

The decline with temperature of the strength of the dry material of the cell wall, as determined by the pressure required for fracture, suggests that methods, such as those described here, may yield data which will contribute to the intimate problems of cell wall structure, particularly when used in conjunction with the rheological analysis suggested earlier Such an approach has been adopted by Treitel for cylindrical plant tissues (8)

Results of the microscopic examination of the sections in polarized light indicated that minute compression failures are not significantly more abundant in the middle zone than below the yield line, which suggests that the plastic deformation in the zone of flow involves micellar or molecular re-arrangement rather than any form of crushing, even on a microscopic scale In this connection it is of interest that another recent paper by Wardrop and Dadswell (11) indicates that, for certain tests carried out at ordinary temperatures under very different conditions from the present ones, short-time loading produced no widespread minute compression failures, whereas there was some development of minute compression failures in beams loaded for long periods However, the stresses were high A point in favour of the assumption of a true form of plasticity in the middle zone of Fig 5 is the well-known plasticizing action of moisture upon wood, which has been discussed quantitatively by Seborg, Millett, and Stamm (7) and briefly by one of the present writers (1)

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The Influence of Fertilizers on Take-All of Wheat

By H. R. Angell, Ph.D. *

Summary.

1. Infection of roots of wheat by root-rotting organisms in differently fertilized plots in a randomized block was not significantly different from the controls.

2. Soil moisture not having been a limiting factor in 1943, the percentage of whiteheads was, in comparison with the controls, significantly lower in the plots manured with a mixture of ammonium sulphate and super-phosphate, with or without trace elements.

3. The effects of some other fertilizers are also noted.

4. Variation in the percentages of plants with whiteheads or incipient whiteheads in different manurial treatments suggests that the economic importance of the disease is significantly influenced by available plant food.

5. Using the control plots as the basis of comparison, the percentages of plants with infected roots in the different treatments are not significantly different, but the percentages with whiteheads or incipient whiteheads are significantly different. The difference between the percentage of root infection and the percentage of whiteheads appears to indicate induced resistance to the development of the disease in the plant.

6. The bearing of these results on the control of take-all, and some factors that may interfere with control are discussed.

1. Introduction

It has been stated by Russell (11) that *Ophiobolus graminis* Sacc., the organism associated with take-all of wheat, *Triticum aestivum* L., is present in the virgin sod in Saskatchewan. Since that time it has become apparent that " the widespread occurrence of take-all as a disease of cereal crops is undoubtedly to be attributed to the cosmopolitan distribution of *O. graminis* as a root disease of grasses " (Garrett, 6). From the results of the writer's experiments and observations, it has been evident that this view concerning the distribution of the organism holds true under the conditions prevailing in the Australian Capital Territory.

In experiments in drums the writer (1, 2) showed that in the second season the disease was of little consequence if plant food was conserved from the first season's crop, but was destructive if plant food was depleted by it. In the experiments, the soil in some of the drums was infested with a culture of the organism in the first year, to other drums nothing was added, but naturally occurring root-rotting organisms were present as was shown by the occurrence of lesions at harvest time. In the first year the disease caused marked under-development of the plants in the artificially infested drums, consequently a larger supply of nutrients was conserved in those drums for the second year's crop. In the drums that were not artificially infested the plant food was nearly exhausted by the crop in the first season, little nutrient remained for the needs of the second season's crop, consequently take-all was destructive.

In a recent paper by White (16) and in the literature reviewed by Garrett (6) it is assumed that control of the disease after fallowing is due mainly to the elimination of root-rotting organisms from the

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soil. The relative efficiency of two years of bare fallow and two years of pasture, respectively, in reducing the percentage of take-all should, however, be noted (16). In the present paper there is evidence that the percentage of plants with infected roots in the differently manured plots does not differ significantly from that in the controls, though there are differences between some of the treatments. The differences in the percentages of plants with whiteheads, however, show that susceptibility to the progress of the disease is conditioned by the nutrients added to the soil (1, 2). The degree of damage to the roots also appears to be influenced by plant food (4, 13). The results of these pot and plot experiments suggest that control of take-all by fallowing may be due to available plant food (3, 8) and not to the elimination of root-rotting organisms.

Extension of the writer's experiments from pots to field plots was facilitated by the cooperation of a wheatgrower on a farm eight miles from the laboratory in Canberra. The disease was destructive in 1939 when wheat was first sown on the area used for this experiment. The history of the field, the soil type, and the distribution of the disease in 1939 and 1940 were discussed by White (15). The experiment here described was conducted in 1943 on a plot in this field in which wheat was grown every season since 1939. White (16) reported that on this area in 1942 the percentage of take-all was 76 and 72 per cent.

The effects of added nutrients on the host, on infection, and on disease development under the conditions prevailing in 1943 are here reported and discussed.

2. Field Experiment—Methods

The usual tillage operatiohs were done and the seed was sown in late autumn as is usual in this area.

The experiment was designed as a randomized block arrangement of four replications with eight plots, each 25 feet by 25 feet, per block. In each block, a mixture of the trace elements listed below was applied to one of the plots and a mixture of the same trace elements and superphosphate to another. These plots were then halved and ammonium sulphate applied to one half. Within each block there was also a control plot to which nothing was added. The remaining five plots per block were given the following treatments:—

>Superphosphate.
>Ammonium sulphate with and without superphosphate.
>Blood manure with and without superphosphate.

Wherever used, the rates of dressing were—

Ammonium sulphate	..	8 cwt./acre.
Superphosphate	..	5 cwt./acre.
Blood manure ..	..	8 cwt./acre.
Zinc sulphate	..	12·0 lb./acre.
Manganese sulphate	..	12·0 lb./acre.
Copper sulphate	..	3·6 lb./acre.
Borax	..	0·6 lb./acre.
Ammonium molybdate	..	0·6 lb./acre.

The growing conditions from seeding to maturity were much better than is usual in this district. The rainfall was well distributed, the monthly totals taken at the experiment station nearby being given below.*

Quadrat samples were taken from each plot in December when the ears could be sharply differentiated as healthy, intermediate, or white-headed. The ears of intermediate type were distinguished by their greyish-green hue, and such ears if left to mature would have produced pinched to very pinched grain. For treatment comparisons it was decided to group them with plants with whiteheads.

The root systems were examined for lesions and rated according to whether lesions were present or absent. Under the circumstances it was impracticable to estimate the amount of root damage, either in the absolute or relative sense. Estimates of the amount of damage to roots and the relation to whiteheads or other signs of take-all in other experiments were dealt with elsewhere (1, 2).

The mean number of ears per plant was recorded as an index of shoot development.

3. Results

The results are given in Table 1. For the ratings of health of roots and ears, the variance between replicates of a treatment and the corresponding mean were approximately proportional, making it necessary to use the square root transformation of the ratings before analysis. The means of the transformed values, together with minimum differences for significance at the 5 per cent. and 1 per cent. level are given. Because of the design, the error appropriate to the estimate of ammonium sulphate effects in the presence of the trace elements with or without superphosphate, is different from the error for the remaining comparisons.

The principal feature of the results shown in the table is the one with which this paper is primarily concerned. It is the generally beneficial effect of the application of a mixture of ammonium sulphate and superphosphate, with or without the trace elements. The beneficial effect of these fertilizer treatments is shown in the significant increase in the number of ears per plant, and the relatively small percentage of whiteheads or incipient whiteheads. By contrast, in none of the plots is the percentage of plants with healthy roots significantly different from the controls. There are, however, three treatments: No. 10—trace elements plus superphosphate plus ammonium sulphate, No. 4—superphosphate plus ammonium sulphate, and No. 5— blood manure, in which the percentage of plants with healthy roots is significantly higher than in two other treatments, No. 2—ammonium sulphate only, and No. 7—trace elements only. With the high percentage of plants with healthy roots a high average number of ears per plant is associated.

In one fertilizer treatment—the trace elements and ammonium sulphate—the low percentage of whiteheads is in marked contrast with the high percentage in the corresponding ammonium sulphate

* January 246 points, February 8 points, March 89 points, April 350 points, May 262 points, June 33 points, July 122 points, August 204 points, September 220 points, October 297 points, November 344 points, December 161 points. Total 2,336 points.

TABLE 1.—SUMMARY OF RESULTS OF FIELD EXPERIMENT.

Treatment	Percentage of Whitehead and Intermediate Types		Percentage of Plants with Healthy Roots		Mean Number of Ears per Plant
	Mean Value	Mean of Square Root Transformed Value	Mean Value	Mean of Square Root Transformed Values	
1. Control	15·60	3·38	15·15	3·85	1·35
2. Ammonium sulphate	13·15	3·12	7·90	2·28	1·64
3. Superphosphate	13·40	3·25	16·72	4·07	1·23
4. Superphosphate plus ammonium sulphate	2·32	0·75	19·25	4·30	2·67
5. Blood manure	12·57	3·13	20·32	4·28	2·10
6. Blood manure plus superphosphate	7·80	2·52	14·15	3·62	1·92
7. Trace elements	46·57	6·73	7·70	2·35	1·16
8. Trace elements plus ammonium sulphate	0·45	0·45	6·85	2·43	1·63
9. Trace elements plus superphosphate	21·30	4·22	10·70	3·05	1·48
10. Trace elements plus superphosphate, and ammonium sulphate	0·40	0·32	33·57	5·42	2·25
Minimum difference for significance at 5 per cent. and 1 per cent. between treatments 7 and 8, or 9 and 10		1·73, 2·61		1·70, 2·58	0·87, 1·32
Minimum difference at 5 per cent. and 1 per cent. between any other treatment pairs		2·41, 4·03		1·92, 3·21	0·65, 1·09

only. The high percentage of whiteheads in the plots receiving only the trace elements is outstanding. The reasons for the divergences cannot be deduced from the present results. Pot experiments have yet to be made to test the effects of these specific mixtures of fertilizers on the soil from this field. For the same reason the trend with blood manure and superphosphate cannot now be discussed. The failure to obtain a satisfactory response from the addition of superphosphate alone is not surprising, because in pot experiments now in progress with this soil no advantage over the controls results from the addition of superphosphate alone.

4. Discussion

In the field experiment dealt with in this paper, it is apparent that infection of the roots of wheat by *Ophiobolus graminis* occurs very generally, almost independently of ordinary fertilizer treatments, indicating that some degree of root infection might be usual. The development of the disease as gauged by the percentage of empty or nearly empty heads is very different, being closely associated with treatment of the soil. The difference between the percentage of plants with root infection and the percentage of plants with diseased heads can therefore be used as an indication of host susceptibility modified by the various soil treatments.

Under the conditions prevailing during the season, in which there was an equitable distribution of the rainfall, the supply of soil moisture was adequate for the needs of the crop, and a well marked degree of control was obtained in the plots in the experimental area to which a mixture of an inorganic nitrogenous fertilizer and super-phosphate was applied. With a comparable soil moisture supply, it is likely that equally good control of take-all would be obtained on the average wheat farm by the natural accumulation of nitrogen in fallowed land, and the usual application of superphosphate. Call (3) has concluded from his experiments in Kansas that " early ploughing in preparing land for wheat appears to be of value under eastern Kansas conditions rather because of the large supply of plant food liberated, especially nitrates, than for any additional moisture that is stored in the soil by the early cultivation. It also appears that poor results are secured from late ploughing chiefly because plant food is not liberated in sufficient quantities to supply the needs of the crop." Richardson and Gurney (8) have shown that under the conditions prevailing at the Waite Agricultural Research Institute, South Australia, the amount of nitrate nitrogen accumulated in the soil by fallowing was sufficient for a prolific wheat crop, a basal dressing of 288 lb. of superphosphate per acre being added at sowing time. They also have stated that soil moisture conservation in the fallowed land was only slightly higher than in stubble land. They also have found that the application of $\frac{1}{2}$ cwt. to 2 cwt. of sulphate of ammonia to stubble land gave profitable increases in the yield of crops sown on stubble. Clark (4) has demonstrated that the degree of control of the root-rotting phase of take-all was correlated with the nitrate nitrogen in the soil, and Stumbo *et al.* (13) have obtained good control of root-rot by the maintenance of available phosphorus and nitrate nitrogen content at suitable levels. In ordinary farming practice in the wheat belt of Australia, a good supply of available plant food in

the soil has been usually ensured by rotation of crops, fallowing, the application of phosphates and sometimes other fertilizers. As has been indicated by the widely varying response of the soils collected from different sources in and near Canberra to fertilizers and amendments, the specific needs of different districts, and soils in those districts in the wheat belt, might eventually be studied with profit.

Instances of failure to obtain control of take-all by the usual cultural methods may be due to naturally poor soils (9), soils exhausted by repeated cropping (7), to competition with weeds (16), to the leaching of available nutrients by heavy rains, to a limiting or sub-optimal supply of soil moisture during the growing season, or to a combination of two or more of these circumstances.

Sewell and Melchers (12), and Griffiths (7) state that repeated cropping of fields to wheat is usually followed by an increase in the economic importance of take-all. Exceptions are recorded by Fellows and Ficke (5), hy the Waite Agricultural Research Institute (14), and in this paper. Some think that destructiveness of the disease is due directly to the building up of inoculum in the soil in the presence of the host plant, but if this were so it would be difficult to account for these exceptions and for the relative freedom from take-all in the continuous wheat plots at Rothamsted (10). The writer's experiments, however, show that impoverishment of the soil by repeated cropping is associated with increased susceptibility to the development of the disease. Clark (4) notes that in successive cropping take-all appears earlier in pots containing small amounts of soil than in those containing larger amounts. If the fertility of the soil is maintained by the regular addition of nutrients, and soil moisture is not a limiting factor*, little if any tendency is noted for the above-ground symptoms of take-all to appear. Soil collected from an area adjoining the site of the experiment here described, in which plants were badly affected with take-all in five successive seasons, was used to grow plants in drums, but the disease did not begin to re-appear until the fourth successive crop in the drums. Freedom from the disease in the drums is shown to be due to adequate water supply that allows the plants to make use of the store of available plant food in the soil. In drums in which the soil moisture, replenished by surface applications, is but little more than sufficient to keep plants growing, the appearance of the plants is the same as in the field in dry years, whiteheads being very common. *Ophiobolus graminis* is not easily isolated, but the cultures obtained indicate that the distribution of the organism is essentially the same in both the well-watered drums, and those that are almost wholly dependent upon rain water. In practice, therefore, control of take-all in areas or years in which soil moisture is not a limiting factor, depends on resistance to the development of the disease associated with proper nutrition.

5. Acknowledgments

* Unpublished work.

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The Effect of Potassium on the Growth of Subterranean Clover and Other Pasture Plants on Crawley Sand

1.—Pot-culture Experiments

By R C Rossiter, B Sc (Agric),* and E H Kipps, B Sc *

Summary.

1 The results are given of two pot-culture experiments with Crawley sand at the Institute of Agriculture, Perth, Western Australia in which responses to potash were obtained in the presence of phosphate with Dwalganup and Mt Barker subterranean clover

2 Wimmera ryegrass grown in association gave responses to potash which were barely significant statistically

3 Potassium deficiency symptoms appeared in the clover species more rapidly in the presence than in the absence of the grass

4 Leaf-area measurements indicated no significant potash effects in the clover up to approximately 60 days from germination Pronounced and significant effects were obtained one month later

1. Introduction

The effect of mineral deficiencies on the growth of pasture plants is of particular importance in the south-west of Western Australia, where light-textured soils constitute the major portion of the area. Because subterranean clover is the most widely used pasture species, the nutritional problems relating to this plant merit special attention. That superphosphate has played an outstanding role in the establishment and productivity of this clover has been recognized for many years (Mursell, 1944) More recently copper deficiency has been demonstrated (Jones and Elliott, 1944)

The investigations herein reported were carried out at the Institute of Agriculture, Perth Profile details of the local soil type—Crawley sand—have been determined by one of the authors (Rossiter, 1938). Allied types occur in the Swan coastal plain (Teakle, 1938), so that the present studies may have more than immediately local application

An experiment conducted in 1941 to examine the effects of various nutrients on a disease of lupins, since shown to be of virus origin (Norris, 1943), resulted in highly significant responses to potassium by subterranean clover which appeared as a volunteer species A second trial in the following year failed to confirm this response The two trials described below formed one phase of a more comprehensive investigation on the potassium problem In the second part of the paper the results of field trials will be given.

2. Methods

Experiment 1

Earthenware pots of 13 5 kg capacity, the inner surface coated with paraffin wax, were used throughout Seed of the Dwalganup variety of subterranean clover (*Trifolium subterraneum* L) was germinated in yellow subsoil sand subsequent to inoculation by the admixture of a small amount of topsoil Four seedlings were transplanted to each pot on May 21, 1943. Soil moisture was maintained

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within the range of 30–40 per cent. of the maximum water-holding capacity (8–10 per cent. actual moisture content). Distilled water was used for this and also the second trial.

Eight treatments were imposed in a 2 x 2 x 2 factorial design with four replications, in a simple randomized arrangement. These treatments may be denoted by: O, P, K, Ca, PK, PCa, KCa, and PKCa, the actual quantities of salts employed being as follows:—

$P-Na_2HPO_4.12H_2O$ .. .. .. 2·7 g./pot*
K- KCl .. .. 1·0 g./pot
$Ca-CaSO_4.2H_2O$ .. .. .. 13·6 g./pot

In addition, a basal nutrient mixture was added, consisting of—

$Cu-CuSO_4.5H_2O$ .. . .. 0·10 g./pot
$Zn-ZnSO_4.7H_2O$ .. .. .. 0·10 g./pot
$Mn-MnSO_4.4H_2O$ .. .. .. 0·17 g./pot
$B-Na_2B_4O_7.10H_2O$ .. .. .. 0·10 g./pot
$Mo-NaHMoO_4$ .. .. 0·03 g./pot

The $CaSO_4$ was applied to the pots 10 days, and the remaining nutrients 1 day, before transplanting.

All plants were harvested at full flowering on September 7, 1943, and the tops oven-dried at 90°C. for 24 hours, for dry weight determinations.

The chemical analyses were carried out at Canberra.

Experiment 2.

Enamelled pots of relatively small capacity (1·50 kg.) were used in the second trial. Two species were tested, viz., subterranean clover (Mt. Barker variety) and Wimmera ryegrass (*Lolium rigidum* Gaud.). The clover seed was inoculated as before, and then sown directly into the pots, and with the grass seed where appropriate, on June 3, 1944. Emergence commenced on June 7, and six days later the seedlings were thinned out to four plants per pot with clover alone, and eight plants per pot—four of each species—where the grass was grown in association.

A basal dressing of $Ca(NO_3)_2$ was applied to all pots on June 16, and the differential nutrient treatments applied on June 19.

Altogether, 18 treatments were involved: the two nutrients, phosphorus and potassium, each at three levels, with and without Wimmera ryegrass grown in association with the clover. The scheme may be represented as follows:—

$$\left.\begin{matrix} P0 \\ P1 \\ P2 \end{matrix}\right\}3 \quad x \quad \left.\begin{matrix} K0 \\ K1 \\ K2 \end{matrix}\right\}3 \quad x \quad \left.\begin{matrix} W0 \\ W1 \end{matrix}\right\}2$$

in which—

$P1-NaH_2PO_4.H_2O$ .. .. .. 0·139 g./pot†
$P2-NaH_2PO_4.H_2O$ .. .. .. 0·278 g./pot
K1-KCl .. .. 0·139 g./pot
K2-KCl .. .. 0·278 g./pot
W1→Wimmera ryegrass in association

* On a surface-area basis 0·68 g./pot is equivalent to 1 cwt./acre.
† 0·139 g./pot is equivalent to 1 cwt./acre on a surface-area basis.

The basal dressing of $Ca(NO_3)_2$ was applied at $0 \cdot 139$ g. per pot to provide available nitrogen for the grass and to ensure an adequate calcium supply for the subterranean clover.

Each treatment was replicated five times in a randomized block lay-out.

On three occasions during the growing season—July 8, August 2, and September 1—total leaf area was estimated on each pot. For this purpose a rating technique (linear scale) was employed, based on a second degree polynomial regression equation relating total leaf area—determined by graphical means—to maximum width of central leaflet. The formula was calculated from measurements on 50 leaves of varying size. This method is similar to that used by Bald for potatoes (Bald, 1943). Although the rating scale was rather difficult to apply, the estimates were easily checked by direct measurement of leaflet width.

Harvesting took place on September 23, shortly before flowering was due to commence in the clover. The tops were carefully separated in the grass/clover mixture. All samples were then oven-dried and weighed as in Experiment 1.

3. Results

Experiment 1.

(a) *Total Dry Weight of Tops.*—A summary of the data is given in Table 1. Despite the wide range in mean values, intra-treatment variances were of the same order of magnitude and no transformation was used prior to analysis.

TABLE 1.—MEAN DRY WEIGHT AND EFFECT—SUBTERRANEAN CLOVER.

Treatment	Dry Weight	Effect
	g /pot	
O	5·03	—
P	9·16	+6·25**
K	5·07	+0·93
Ca	3·53	−2·01**
PK	13·34	+1·45*
PCa	9·00	+0·05
KCa	2·46	−1·17*
PKCa	9·58	+0·62
Standard error	±0·787	±0·557

** Significant at 1 per cent level   * Significant at 5 per cent level

The pronounced response to phosphorus was expected, and confirms earlier field-plot experiments. However, the depressing effect of $CaSO_4$ was quite unexpected; it will be discussed further at the end of the paper.

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Examination of the interaction effects shows barely significant results for P x K and K x Ca. Of these the former was positive, suggesting that the effect of potassium was greater at high than at low levels of phosphorus; from the latter it appears that the depression in yield arising from CaSO₄ was greater in the presence than in the absence of potassium.

It should be mentioned here that a subsidiary contemporaneous experiment showed a significant depressing effect of the basal solution in the absence of phosphorus. This was demonstrated to arise from excess boron. Whether, as a consequence, interaction in the main trial was affected is not known.

(b) *Mineral Content of Tops.*—In Tables 2 and 3 are shown the results of these analyses; both absolute and percentage contents are presented.

TABLE 2.—RELATIVE AMOUNT (G./100 G.) OF MINERALS AND SOLUBLE ASH IN TOPS OF SUBTERRANEAN CLOVER.

Treatment	Phos-phorus	Potas-sium	Calcium	Mag-nesium	Sodium	Nitrogen	Sulphur	Soluble Ash
O	0·18	1·19	0·96	0·33	1·01	2·27	0·28	7·28
P	0·32	0·70	0·66	0·30	1·52	2·41	0·20	6·68
K	0·18	2·11	0·92	0·30	0·70	2·32	0·29	8·36
Ca	0·20	1·42	1·35	0·48	0·89	2·43	0·89	9·51
PK	0·31	1·40	0·71	0·30	1·11	2·41	0·20	7·20
PCa	0·31	0·79	1·16	0·44	1·24	2·33	0·82	8·46
KCa	0·18	2·53	1·26	0·45	0·50	2·57	0·91	10·06
PKCa	0·27	1·92	1·01	0·35	0·73	2·52	0·73	8·01

TABLE 3.—ABSOLUTE AMOUNT (MG./POT) OF MINERALS AND SOLUBLE ASH IN TOPS OF SUBTERRANEAN CLOVER.

Treatment	Phos-phorus	Potas-sium	Calcium	Mag-nesium	Sodium	Nitrogen	Sulphur	Soluble Ash
O	9	60	48	17	51	114	14	366
P	29	64	60	27	139	221	19	612
K	9	107	47	15	35	118	15	424
Ca	7	50	48	17	31	86	31	336
PK	41	187	94	40	148	321	27	961
PCa	28	71	103	40	112	210	74	761
KCa	4	62	31	11	12	63	22	247
PKCa	26	184	97	33	70	241	70	267

Two general types of response can be distinguished:—

(1) Where addition of nutrient X tends to give a considerable increase in the relative amount of that nutrient. Where this addition also leads to an increase in yield there is obviously a relatively greater

increase in absolute amount of X (e.g. phosphorus O — P, K — PK, Ca — PCa, KCa — PKCa; potassium P —PK, PCa — PKCa). Where there is no effect on yield, the increase in absolute amount is naturally proportionate (e.g. potassium O — K, and as border line cases PCa — PKCa, Ca — KCa; calcium P — PCa). Where the addition of nutrient X actually decreases yield, the absolute amount of X may change only slightly (e.g. potassium Ca — KCa; calcium O — Ca, PK — PKCa) or it may be actually depressed (e.g. calcium K — KCa).

(2) Where addition of nutrient X may have a variety of effects on the relative and absolute amounts of nutrient Y. Where growth is increased by X there is a tendency for the relative amount of Y to be decreased. This is doubtless a partial explanation of the effects of phosphorus on the relative amounts of potassium, calcium, magnesium, sulphur, and soluble ash. The effect on potassium, however, is complicated by the fact that phosphorus was applied as Na_2HPO_4—the interaction diagrams for relative amounts of potassium and sodium are almost exact mirror images—and high sodium in the medium has depressed the intake of potassium.

The effect of Ca on relative amount of magnesium is striking. (In this instance growth is decreased by X and the relative amount of Y increased.) A similar example of positive correlation between calcium and magnesium percentages has been reported for lucerne (Hunter *et al.*, 1943).

The effect of P on total nitrogen content is also of interest. In the absence of Ca, P resulted in an increase in relative amount of nitrogen, which is in keeping with other work on legumes (e.g. Teakle, 1945). However, the fact that the relative amount of nitrogen was decreased with P in the presence of Ca is suggestive of toxic effects of the latter.

Since this paper is primarily concerned with potassium nutrition, it is advisable to emphasize the importance of the decrease in relative content of potassium when phosphorus is applied (both in the presence and absence of Ca). In the absence of K the increased yields with P caused a slightly greater absolute amount of potassium in the tops.

Experiment 2.

(*a*) *Total Dry Weight of Tops.*—In this and the following section a logarithmic transformation has been used wherever the data are treated statistically.

(1) *Subterranean clover.*—Highly significant treatment effects were obtained for P, K, W, PK, and PW. A summary of the data is given in Table 4.

The significant depressing effect of the associated grass on the yield of clover was, of course, to be expected. Of some interest also is the P x W interaction. Examination of the data will show that the mean relative decrease in yield due to Wimmera ryegrass was significantly higher at P0 than at P1 and P2. Taken in conjunction with the evidence on Wimmera ryegrass in Table 5, this probably reflects a differential species response to phosphorus, the clover responding more markedly than Wimmera ryegrass.

TABLE 4.—MEAN DRY WEIGHTS (G./POT) OF SUBTERRANEAN CLOVER, AS
AFFECTED BY SUPPLY OF PHOSPHORUS AND POTASSIUM AND
ASSOCIATED GROWTH OF WIMMERA RYEGRASS.

Without Wimmera (W0).

—	P0	P1.	P2	Mean
K0 ..	1·207 (2·0818)	2·631 (2·4202)	2·542 (2·4052)	2·006 (2·3024)
K1 ..	1·416 (2·1510)	3·314 (2·5204)	3·286 (2·5166)	2·489 (2·3960)
K2 ..	1·385 (2·1412)	3·399 (2·5314)	3·391 (2·5304)	2·518 (2·4010)
Mean ..	1·332 (2·1247)	3·095 (2·4907)	3·049 (2·4841)	2·326 (2·3665)

With Wimmera (W1).

—	P0	P1	P2	Mean
K0 ..	0·517 (1·7138)	1·336 (2·1256)	1·172 (2·0690)	0·932 (1·9695)
K1 ..	0·511 (1·7084)	1·675 (2·2240)	1·589 (2·2010)	1·108 (2·0445)
K2 ..	0·472 (1·6740)	1·792 (2·2532)	1·715 (2·2342)	1·132 (2·0538)
Mean ..	0·500 (1·6987)	1·588 (2·2009)	1·472 (2·1681)	1·053 (2·0226)

Average W0 *and* W1.

—	P0	P1	P2	Mean
K0	0·790 (1·8978)	1·875 (2·2729)	1·726 (2·2371)	1·368 (2·1359)
K1 ..	0·850 (1·9297)	2·356 (2·3722)	2·284 (2·3588)	1·661 (2·2202)
K2 ..	0·808 (1·9076)	2·468 (2·3923)	2·412 (2·3823)	1·689 (2·2274)
Mean ..	0·816 (1·9117)	2·217 (2·3458)	2·118 (2·3261)	1·565 (2·1945)

Standard errors   mean of   5—(0 0229)
10—(0 0162)
15—(0 0132)
30—(0 0093)

Note — The non-bracketed figures are geometric means of the original observations   Figures
in parentheses are mean log x 10¹

As in Experiment 1 the response to phosphorus was highly sig-
nificant. However, at the P2 level, yields were slightly, though sig-
nificantly, less than at P1.

The P x K interaction is exhibited more clearly in this trial than
in the first. In the absence of phosphorus no beneficial effects of any
magnitude followed on the application of potassium. In its presence
yields were increased significantly over and above those obtained with
phosphorus only. Averaging P1 with P2 and K1 with K2, the actual
and relative mean yields were—

	Control.	P only.	K only.	P + K.
	0·865	1·920	0·942	2·531
	100	222	109	293

Bearing in mind the greater variability in the first trial, and also
differences in strain and time of harvest, the relative yields for each
experiment are in close agreement.

(2) *Wimmera ryegrass.*—Mean values are given in Table 5. The trends are obviously similar to the trends of subterranean clover yields. The interaction component is significant in the 2 x 2 table formed by K0P0, and the means obtained by grouping K1 with K2 and P1 with P2. With increased amounts of P at all levels of K there is an increased yield. On the other hand there is a significant response to K only in the presence of P1 and P2.

TABLE 5.—MEAN DRY WEIGHT (G./POT) OF WIMMERA RYEGRASS AS AFFECTED BY PHOSPHORUS AND POTASSIUM SUPPLY.

				Mean
K0	0·720 (1·8562)	0·905 (1·9568)	1·085 (2·0354)	0·890 (1·9495)
K1	0·773 (1·8882)	1·212 (2·0832)	1·504 (2·1472)	1·095 (2·0395)
K2	0·705 (1·8484)	1·275 (2·1054)	1·335 (2·1252)	1·063 (2·0263)
Mean	0·731 (1·8642)	1·118 (2·0485)	1·267 (2·1026)	1·012 (2·0051)

Standard errors mean of 5—(0 0331)
1) (0 0191)
Note Figures in parentheses are log × 10² as in Table 4

(b) *Changes in Leaf Area of Subterranean Clover.*—Since a large number of pots are required for the determination of changes in dry weight with time, especially for a large number of treatments, it was decided to use leaf areas as indices of ontogenetic change. Total leaf area on any given occasion is an index of the capacity of the plant to produce dry matter in the immediate future. In early stages of growth, however, there tends to be a strong correlation between leaf area and total yield, though this may not be relied upon to the extent of using the former as a strict index of the latter. In any case, leaf area data are of considerable value on their own account.

In the present trial measurements were made on three occasions. Results are shown in Fig. 1.

On the first occasion, 41 days after germination, the P treatments only showed highly significant differences; both P1 and P2 were greater than P0, the increase being of the order of threefold. The main effect of P therefore took place prior to this occasion.

At day 57 this increase with P was almost identical in magnitude, whilst the associative effect of Wimmera ryegrass had now produced a highly significant depression in leaf area.

The P and W responses were still predominant at day 87, but highly significant effects due to potassium and its interaction with P and W (both singly and together) had now become obvious. It should be mentioned that at day 57 a barely significant P x W interaction was detected, the decrease in leaf area with Wimmera present being greater at P2 than at P0. The day 87 figures indicate that this was influenced largely by the relatively great difference at K0.

Stated in another way, the response to potassium with phosphorus present was relatively greater when the clover was grown in association with Wimmera ryegrass.

This second order interaction appears to be due entirely to an effect on leaf area, as such. Analysis of the data for total number of leaves at day 87 showed no effect of either K or P x K, although a significant increase and decrease to P and W respectively. Premature senescence of the older leaves accounted for the lack of increase in total leaf areas between t_2 and t_3 in P1W1 and P2W1; the fact that the P x K x W interaction for total dry matter was not significant may be at least partially accounted for, then, in terms of higher leaf dry weight to area ratios in the treatments mentioned.
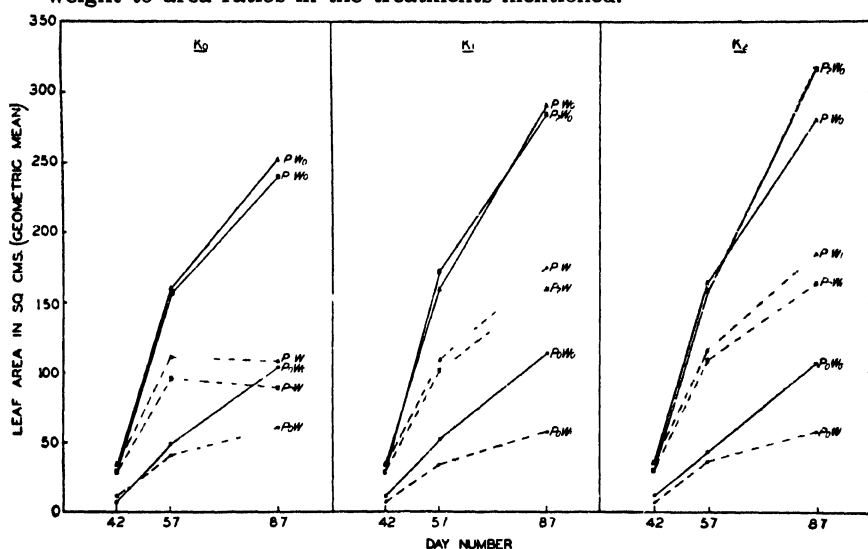
FIG. 1.—Showing the effect of potash, phosphorus, and presence and absence of Wimmera ryegrass on the changes in total leaf area of subterranean clover.

Relative leaf-growth rates were calculated using the equation

$$R_{LA} = \frac{\log e\ L_{A_2} - \log e\ L_{A_1}}{t_2 - t_1},$$ where L_A = leaf area in sq. cm., and t = time in days.

TABLE 6.—RELATIVE LEAF GROWTH RATES (SQ.CM./SQ.CM./DAY) OF SUBTERRANEAN CLOVER, AS AFFECTED BY PHOSPHORUS AND POTASSIUM SUPPLY, AND ALSO ASSOCIATED GROWTH OF WIMMERA RYEGRASS.

		Occasion 1–2			Occasion 2–3.		
		K0	K1	K2	K0	K1.	K2.
P0W0		0·104	0·107	0·098	0·028	0·027	0·030
P0W1	.	0·081	0·088	0·089	0·017	0·018	0·014
P1W0	..	0·106	0·107	0·106	0·015	0·020	0·018
P1W1	..	0·082	0·086	0·082	0·001	0·016	0·015
P2W0	..	0·106	0·114	0·108	0·014	0·017	0·023
P2W1	..	0·076	0·085	0·092	0·002	0·115	0·014

Relative leaf growth rates were everywhere depressed by Wimmera ryegrass, the depression being relatively greater between t_2 and t_3: the latter would be in keeping with progressive competition between grass and clover.

The effects of both phosphorus and potassium were only slight between t_1 and t_2. Between t_2 and t_3, however, there was a fairly consistent decrease in relative leaf growth rate from P0 to P2; in the presence of Wimmera ryegrass this was extremely pronounced at K0, yet inappreciable at K2. Although the P1K0W1 and P2K0W1 figures were determined primarily by premature leaf senescence, the general effects of the phosphorus treatments is not so readily understood. No satisfactory explanation can be put forward.

4. Discussion

The first point arising from the data has to do with the depressing effect of high $CaSO_4$ levels on growth. Although it is conceivable that the high relative amount of calcium may have reduced phosphorus uptake as suggested by the work of Shih (see Richards, 1941), a more likely explanation is that toxic effects were produced. It will be noted (*vide* Table 2) that the total sulphur content in the $CaSO_4$ treatments was of the order of 0·8 per cent. (on a dry matter basis). Since the actual sulphate sulphur constituted almost 80 per cent. of this amount, it may well be that the plants were adversely affected by toxicity of the SO_4 ion. Eaton (1942) has shown that such toxic effects may depress growth gradually without any obvious external symptoms*. There is one further possibility, viz., that soil pH was reduced sufficiently to affect plant growth. Supporting data are scanty: it has been shown that $CaSO_4$ at the rate of 30 cwt. per acre, in the absence of phosphorus, will reduce the pH of Crawley sand from 6·2 to 5·3.

That a P x K yield interaction occurs with subterranean clover on the soil herein studied appears reasonably well established; indeed, further evidence on this point will be presented in Part 2 of this paper. No such interaction was present in the leaf-area data of experiment 2 up to approximately 60 days from germination, so that potassium supply presumably was not limiting during early growth.

Now the exchangeable K in the Crawley sand is of the magnitude of 0·06 m.e. per 100 g., so that the total exchangeable K per pot for experiments 1 and 2 was 316 mg. and 35 mg. respectively. For the former trial the quantity of exchangeable soil potassium should have been more than adequate to account for the absolute potassium content of the highest yielding treatment (PK), and in the second trial, almost adequate. The following tentative explanation is therefore put forward. During the first few weeks, when the growth rate was low, the rate of supply of exchangeable K from the soil was adequate for normal metabolic functions, even where P was not limiting. At a later stage, when the growth rate was relatively higher on all treatments, the rate of supply of soil K became limiting with high P but not with low P. Of the two nutrients, P was more deficient in the soil than K.

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* It is of interest to calculate the relative SO_4 content of lucerne—the only legume studied—in relation to yield depressions. With a 28 per cent. reduction (on the first cut) the SO_4 content was of the order of 1·2 g./100 g. dry matter. This figure was exceeded in experiment 1.

The position with respect to Wimmera ryegrass is less precise. In the second experiment reported no significant P x K interaction was detected.* This, of course, may have been influenced by the associated effect of the legume, yet the likelihood of a difference in pattern of reaction in the case of grasses is suggested by a pot-culture experiment carried out in conjunction with experiment 1. Perennial veldt grass (*Ehrharta calycina* Sm.) was subjected to the following treatments:—N, P, K, and Ca in a 2^4 factorial design. The only significant effects on total yield were N, P, and the N x P interaction. Chemical data suggest that the grass is capable of tolerating a lower relative amount of potassium than subterranean clover without serious detriment to growth; the mean potassium content of the N–No K plants at the flowering stage was 0.71 per cent. Nevertheless, P x K interactions may occur in grass species, as the work of Verma (quoted by Gregory, 1937) has clearly demonstrated.

A description of the potassium deficiency symptoms observed in the experiments is not intended, since a section of the second part of this paper will be devoted to a comprehensive account of this aspect of the problem.

5. Acknowledgments

The authors are grateful to Mr. E. A. Cornish, Officer-in-Charge, Section of Statistical Mathematics, C.S.I.R., for carrying out much of the statistical work involved in the second trial, and also to Mr. R. F. Williams, Division of Plant Industry, C.S.I.R., for criticism and helpful advice.

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* By grouping the two phosphate levels, P1 and P2, the P x K interaction was found to be significant, though barely so.

The Effect of Potassium on the Growth of Subterranean Clover and Other Pasture Plants on Crawley Sand

2.—Field-plot Experiments

By R. C. Rossiter, B.Sc.(Agric.) *

Summary.

1. The results of field-plot trials, with potash fertilizers, are presented and discussed, and deficiency symptoms in subterranean clover are described in detail.

2. Of the species examined, only one—subterranean clover—showed clearly defined responses to potassium. Wimmera ryegrass responses were small and of doubtful significance, whilst the W.A. lupin, perennial veldt grass, and capeweed, showed either no increase in yield or a slight depression through inter-specific competition with the clover.

3. Seed production in subterranean clover was adversely affected by low potassium supply.

4. Potassium-deficient leaves of subterranean clover showed a high relative content of sodium, but no excessive concentration of calcium, despite the high exchangeable Ca status of the soil type studied

1. Introduction

Evidence from pot trials has been presented of increased yields of subterranean clover (*Trifolium subterraneum* L.) with potassium, in the presence of phosphate on Crawley sand (Rossiter and Kipps, 1947). Furthermore, the association of an annual grass Wimmera ryegrass (*Lolium rigidum* Gaud.) did not materially affect the magnitude of this response, although deficiency symptoms in the legume appeared earlier.

The range of plant species is rather wider in the present trials— a point of obvious importance—yet it should be borne in mind that inter-specific competitive effects may have masked real potassium effects on the species. The point is of rather academic interest, since under field conditions subterranean clover almost without exception is the basic constituent of sown pastures in the south-west of Western Australia.

The potassium deficiency symptoms are described fully with the belief that they will enable recognition of potassium deficiency in subterranean clover elsewhere in southern Australia.

2. Preliminary Trial

(i) *General.*

Descriptions of experimental procedure for each individual trial would occupy undue space, and general methods can, in any case, be inferred from the detailed information of the main trial (see below). All trials were carried out in small plots whose dimensions varied from 10 links by 10 links up to 30 links by 15 links, each trial being limited to a very small acreage. To determine seed yield in subterranean clover, duplicate square link soil samples were taken from each plot to a depth of 3 in., the material sieved, and burr formed

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during the current season carefully separated from the residue. Seed yields were calculated from the regression equation of seed weight on burr weight. Although it is recognized that the sampling ratio was extremely small, significant differences were found in a number of cases.

Experiment 1 was carried out in 1941 and had as its primary objective the study of the effects of potassium, copper, and chromium, in a 2 x 2 x 2 factorial design, on the incidence of a disease in the W.A. lupin (*Lupinus pilosus* Murr. var. *Cosentini* Guss.). Towards the end of the growing season, treatment effects were observed in the volunteer pasture of Dwalganup subterranean clover/Wimmera rye-grass which established beneath the lupin crop. The pasture was therefore harvested for yield determinations.

In Experiment 2, conducted in 1942, the effect of varying levels of potassium was studied on a mixed Dwalganup subterranean clover/Wimmera ryegrass pasture established in 1940 and top-dressed in 1940 and 1941 with superphosphate only. In contrast to the first trial, fertilizers were applied without early surface cultivations and in consequence effective germination occurred earlier.

After analysis of the results of Experiments 1 and 2, it was decided that two further trials should be conducted on the same sites, but with reversed pre-fertilizer treatment. Experiment 3 occupied the original site of Experiment 1, with the differences (a) that a new set of potassium treatments was allotted at random to the previous Cu and Cr treatments (to which no yield responses were observed in 1941), and (b) that no soil cultivation treatments were imposed. The trial was conducted in 1944, having remained open to uniform continuous grazing during 1943.

Experiment 4 was carried out on the site previously occupied by Experiment 2, and in the same year as the third trial, viz., 1944. It differed, again, in two respects from Experiment 2: (a) a new set of potassium treatments was applied to all plots by making use of the split-plot technique, and (b) the whole experimental area was rigorously cultivated prior to application of the potash fertilizers.

In each of the four trials superphosphate was applied at the rate of 2 cwt. per acre as a uniform basal dressing.

(ii) *Results.*

(a) *Herbage Dry Weight Yields.*—In Experiment 1 no differences resulting from the application of either copper or chromium were significant. Potassium, however, produced a highly significant response in the case of subterranean clover, whilst with Wimmera ryegrass the increase in yield was barely significant at the 5 per cent. level (see Table 1). The yields of lupins and miscellaneous species were significantly depressed, presumably by competitive effects of the clover.

The relative magnitude of the response of subterranean clover was much the same in Experiments 1 and 3 in spite of the high yields of miscellaneous species in the latter.

The response of Wimmera ryegrass, though consistent in each experiment, was not significant even at the 5 per cent. level in Experiment 3. Miscellaneous species—chiefly capeweed—showed an insignificant reduction in yield with potassium in the third trial. Expressed

as a percentage of total yield, however, capeweed was diminished from 53 per cent. to 41 per cent. by potassium, this reduction being significant at the 1 per cent. level.

TABLE 1.—MEAN YIELDS OF DRY MATTER (CWT./ACRE).
($K = KCl$ at 1 cwt./acre.)

Species Group	Experiment 1 (1941)			Experiment 3 (1944)		
	No K	K	S E	No K	K	S E
Subterranean clover ..	15·02	25·35	1·115	6·33	9·70	0·649
Wimmera ryegrass ..	1·27	2·01	0·177	3·22	3·83	0 417
Miscellaneous species*	2·45	1·13	0·216	13·35	11·37	0·923
Lupins	82·18	69·88	2·644	.	..	..
Total, minus Lupins	18·74	28·49	1·151	22·90	24·92	0·857

* Miscellaneous species in 1941—*Trifolium procumbens* L., *Tunica prolifera* L., *Briza maxima* L. In 1944—*Cryptostemma calendulaceum* (L.) R. Br., *Vulpia Myuros* L., *Ehrharta longiflora* Sm.

In Experiment 2 no significant responses to potassium were detected, the mean yields for subterranean clover and Wimmera ryegrass being $9·6 \pm 1·48$ cwt. per acre and $11·8 \pm 1·71$ cwt. per acre respectively.

Analysis of the split-plot latin square design of Experiment 4 revealed highly significant main plot differences for " columns," and also a barely significant increase in yield of subterranean clover due to potassium applied to the sub-plots in 1944. The relative responses of the clover to the fertilizer in the several " columns " were as follows:—

A	B	C	D	E
+ 41%	+ 25%	+ 5%	− 5%	+ 4%

This soil fertility trend was not apparent in the 1942 data, and it is possible that the cultivation treatments of 1944 influenced the nature of the responses to potassium. In any case, it may be noted that the magnitude of the response in " column " A is comparable with those of Experiments 1 and 3.

(b) *Seed Yields of Subterranean Clover.* —Analysis of data obtained from Experiment 3 indicated a highly significant effect of potassium on seed production. The relevant yields (together with total tops) were·—

	No K	K	S E
	cwt./acre	cwt./acre	
Seed	2·87	3·96	0·212
Herbage	6·33	9·70	0·649

The relative increase in seed yield was therefore not as pronounced as that in herbage. Whether this difference was real is not known, but the errors in sampling technique for seed production may conceivably account for part of the discrepancy. In this regard it may be pointed out that seed yields, even in the presence of potassium, were low: figures of the order of 10 cwt. per acre are not infrequently obtained on Crawley sand with this clover.

A more comprehensive analysis of the effects of acute potassium deficiency on clover seed is shown in Table 2. This information was collected from a separate experiment, not previously considered.

TABLE 2.—THE EFFECT OF EXTREME POTASSIUM DEFICIENCY ON SEED AND BURR CHARACTERS IN DWALGANUP SUBTERRANEAN CLOVER.

Variate	No K	K
No. burrs per sq. link	40·3	221·2
Total burr wgt.—g /sq link	1·3	13·6
Wgt. per burr—mg	32	68
Seed yield—cwt /acre	1·2	12·1
Wgt. per seed—mg	5·2	8·5
No. seeds per burr	2·8	3·5
Ratio seed wgt to burr wgt	0·46	0·45

The striking decrease in seed yield on the No K plot can be accounted for largely by the smaller number of burrs produced. Although the mean weight per burr under potassium deficiency was approximately one-half that of the plants on the manured plot, the ratio of seed weight to burr weight was almost unaffected by potassium supply. The constancy of the ratio is determined by the smaller weight per seed and the lower number of seeds per burr under potassium-deficient conditions. (Confirmation of the reduction in weight per seed was obtained from Experiment 3 where K and No K values were 6·6 mg. and 5·0 mg. respectively.)

3. Main Trial

(i) *Procedure.*

The site was cleared from virgin forest in 1943 and twice cultivated after the opening autumn rains of 1944 to provide a satisfactory seed-bed. The experiment area was pegged out on May 10, 1944, and comprised 90 plots each 10 links square. A seeds mixture of Dwalganup subterranean clover and perennial veldt grass (*Ehrharta calycina* Sm.) at the rates of 12 lb. per acre and 4 lb. per acre respectively was sown uniformly over the area, using sawdust as a matrix to provide adequate bulk and even distribution. A shallow soil covering was ensured by hand raking.

Seedling emergence was first observed on May 23.

Eighteen treatments were distributed in a factorial randomized block design with five replications, the treatment scheme being as follows:—

$$\left.\begin{matrix} P0 \\ P1 \\ P2 \end{matrix}\right\}3 \quad \text{x} \quad \left.\begin{matrix} K0 \\ K1 \\ K2 \end{matrix}\right\}3 \quad \text{x} \quad \left.\begin{matrix} M0 \\ M1 \end{matrix}\right\}2$$

where—

P1		2 cwt./acre superphosphate
P2	.	. 4 cwt./acre superphosphate.
K1	..	.. 1 cwt./acre KCl.
K2	..	.. 2 cwt./acre KCl.
M1	..	. $\left\{\begin{matrix} 15 \text{ lb./acre } MgSO_4. \\ 5 \text{ lb./acre } CaSO_4. \\ 5 \text{ lb./acre } ZnSO_4. \end{matrix}\right.$

All fertilizer treatments were applied on June 30, 1944, the manures being mixed previously with yellow subsoil sand to provide uniform distribution.

Since the growth of clover was poorer than expected at the conclusion of the first season, yield estimations were made by eye on a 0–10 rating basis.

No fertilizers were applied in 1945. The plots were sampled at the end of this season with a hand-operated shearing machine, using a 25 sq. link sample per plot. Harvested material was air-dried and stored prior to hand separation for estimation of botanical composition and yield of components. Dry weights were obtained by the usual method of oven-drying at 95°C. for 24 hours.

(ii) *Results.*

(*a*) *1944.*—No differences of any consequence appeared until ten weeks after germination (July 28), when phosphate effects were easily distinguished. After a further four weeks (viz., at the end of August) differences due to potassium were noted in the growth of subterranean clover, which by this time had reached the flowering stage. All treatment effects became progressively marked throughout September, and estimated productivity was determined at the end of this month.

Neither perennial veldt grass nor miscellaneous species—chiefly *Tunica prolifera* (L) Scop., *Briza maxima* L., and *Hypochaeris glabra* L.—showed any response to fertilizer treatments; the data for Dwalganup subterranean clover only are presented in Table 3 below.

TABLE 3.—MEAN PRODUCTIVITY RATINGS—DWALGANUP SUBTERRANEAN CLOVER—SEPTEMBER 30, 1944.

	P0	P1	P2	Mean
K0 ..	0·50	2·90	2·55	1·98
K1 ..	0·55	7·30	8·70	5·52
K2	0·55	7·70	9·00	5·75
—	—	—	—	—
Mean .	0·53	5·97	6·75	4·42

s L main effects—0·2006

Treatment variances were relatively constant, and the analysis was performed on the original data. Since the mixed fertilizer treatment produced extremely small and insignificant effects, the summary table relates only to phosphorus and potassium, in which main effects and P x K interaction were all highly significant. This latter finding is in substantial agreement with results previously reported (Rossiter and Kipps, 1947) using the pot-culture technique. Superphosphate alone increased yields from five- to six-fold, whilst potassium, in addition to superphosphate, produced an increase in yield of the order of three-fold compared with the P–No K treatments. Obviously in this trial the area selected was acutely deficient in potassium (cf. data of Table 1).

Mention should be made of occasional localized effects within plots. A number of instances were noted on portions of P and PM plots where the production of clover was abnormally high and the plants without

symptoms of potassium deficiency. Such areas were associated with old tree stump sites and have been frequently observed on Crawley sand. Anderson (1942) has reported somewhat similar effects from wood-ashes in the Meadows district of South Australia, which were attributed to molybdenum. In the present instance unpublished data suggest that the effect is due almost solely to potassium contained in the wood-ash.

(*b*) *1945.*—Shortly after germination (May 1) of the subterranean clover in the second year, it was observed that the legume was almost absent on the No P plots, and that seedling density on the P–No K plots was very much lower than where both fertilizers had been applied in 1944. The former discrepancy in seedling establishment explains the extreme difference in yield between No P and P–No K treatments (see Table 4); the second is in keeping with the results shown in Table 2.

Differences between P and PK treatments were clearly obvious in the clover at the commencement of flowering—early August—and, as in 1944, became progressively greater thereafter.

TABLE 4.—MEAN YIELDS OF DRY MATTER (CWT./ACRE)—OCTOBER 14, 1945.

(*Average of M and No M treatments.*)

Treatment	Subterranean Clover	Perennial Veldt Grass	Miscellaneous Species	
0	trace	3·18	0·67	3·85
P1	2·62	8·26	0·53	11·41
P2	2·11	8·58	0·72	11·41
K1	trace	2·58	0·93	3·51
K2	0·07	2·70	0·90	3·67
P1K1	5·93	7·87	0·33	14·13
P1K2	6·55	7·25	0·30	14·10
P2K1	10·81	8·16	0·28	19·25
P2K2	12·02	8·82	0·37	21·21

The beneficial effects of phosphate on perennial veldt grass were shown throughout the growing season. In the previous paper (Rossiter and Kipps, 1947) it was stated that a highly significant N x P interaction occurred with this grass in a pot-culture experiment; in actual fact there was practically no response to phosphorus in the absence of applied nitrogen, yet in its presence the yield was increased by approximately 30 per cent. Since there is adequate observational evidence that even a single year's growth of subterranean clover increased markedly the yield of non-leguminous pasture species in this State, the differential response of *Ehrharta calycina* to phosphate in the two seasons 1944 and 1945 of the present trial may conceivably be accounted for by an increased soil N status following on the growth of clover in the first season (1944). Furthermore, the absence of any response to potassium in either year of this main experiment confirms evidence from the pot-culture trial already referred to.

Low yields for the miscellaneous species group with the PK treatments reflect competition from the perennial grass and clover.

The three botanical separation groups already considered are explanatory of the " total " column of Table 4. One point of interest may be mentioned briefly: with superphosphate alone the clover contributed only 20 per cent. of the total yield, whereas with phosphate plus potassium—more particularly the higher level of the former fertilizer—the figure was increased to approximately 50 per cent.

During the 1945 season, treatment replicates were more variable than usual with trials on Crawley sand—a fact which may have been due, in no small degree, to a combination of two factors: (*a*) small plot size, and (*b*) recency of clearing. In any case the original data were transformed for purposes of statistical analysis.

TABLE 5.—THE INTERACTION EFFECT OF SUPERPHOSPHATE AND POTASSIUM ON DWALGANUP SUBTERRANEAN CLOVER AND PERENNIAL VELDT GRASS—1945.

$$\text{Variate}\begin{cases} \text{Clover} = \log((\log x + 1) + 1). \\ \text{Veldt grass} = \sqrt{\bar{x}}. \end{cases}$$

Subterranean Clover.

	P0	P1	P2	Mean
K0	..	0·3536	0·3118	0·2218
K1	.	0·4546	0·4992	0·3179
K2	0·0310	0·4616	0·5022	0·3316
Mean	0·0103	0·4233	0·4377	0·2905

S.E. main effects—0·0124.
Body of table—0·0215.

Perennial Veldt Grass.

	P0	P1	P2	Mean.
K0	6·070	10·087	10·193	8·783
K1	5·380	9·917	10·059	8·452
K2	5·612	9·187	10·454	8·418
Mean	5·687	9·730	10·235	8·551

S.E. main effects—0·321.
Body of table—0·556.

Analysis of variance for each set of data demonstrated that the mixed fertilizer produced no significant effects, thus confirming the 1945 observational evidence and also the 1944 results; they are not shown separately in either Table 4 or 5.

For subterranean clover, the main effects for both P and K were significant at the 1 per cent. level, and the P x K interaction significant at the 5 per cent. level. Despite the fact that the increase in yield from P1 to P2 in the presence of K was not significant on the transformed data, it is difficult to conceive that the increase was not actually a real one.

Similarly with *Ehrharta calycina* there was a consistent increase from P1 to P2, which did not reach significance in the analysis of variance. Moreover, the depression in yield with K1 is suggestive of a real competition effect from the clover, even though the effect could not be substantiated by statistical analysis. However, there is no question of the significance of the previously mentioned increase in yield of perennial veldt grass to phosphate.

4. Diagnosis of Potassium Deficiency in Subterranean Clover

(i) *Description of Deficiency Symptoms.*

The following details apply specifically to the Dwalganup strain; Mt. Barker has been examined only cursorily, but appears to follow similar lines.

Where the deficiency is not acute, external symptoms are rarely exhibited until shortly before the flowering stage; in the case of mild or incipient deficiency, symptoms may be delayed almost until maturity. The first sign of deficiency is a slight bronzing of the upper portion of the older leaflets, i.e., above the crescent. Very shortly afterwards—sometimes coincidently—reddish-brown spots appear on the leaf surface; these are aptly described as " stained " areas, and are frequently 1 mm. to 3 mm. in diameter. Closer inspection of these areas usually reveals the presence of a central area of surface " pitting " which seldom becomes extensive. Gradually, the leaf surface becomes chlorotic, particularly above the crescent, and marginal necrosis or " die-back " follows.

Plants exhibiting mild deficiency symptoms appear almost normal in a number of respects, viz., leaf area, tillering, and growth rate. In this they resemble potassium deficiency as described by Richards and Templeman (1936) in the barley plant. Reduction in dry weight then, according to these authors, depends on—

     (1) reduction in leaf area—by early death of leaves; and

     (2) much reduced photosynthetic activity of living leaves.

Decreases in total dry weight not infrequently may be exceedingly difficult to demonstrate in subterranean clover, especially where symptoms are of the incipient type and consequently tardy in development.

Where potassium deficiency is acute, symptoms differ in a number of respects from those described above. Symptoms may be observed as early as six weeks after germination—very rarely before this time; they commence with an ashy-grey to reddish-brown " pitting " on the surface of the older leaflets, the " pitted " areas measuring from 0·5 to 1 mm. in diameter. The necrotic tissue so produced conforms closely in anatomical characters to that described for red clover (Lindenbein, 1936). The leaves in which " pitting " occurs may be normal in colour, or even a slightly darker green than normal, but quickly develop a mild yellowish-green chlorosis.

The most striking character of acute deficiency appears in the youngest leaves developed very shortly after the above-mentioned symptoms have become well-defined. No obvious reduction in leaf-area is noticed in the older affected leaves, yet these young leaves are greatly reduced in size. Moreover, they are dark green in colour, possess a

puckered surface, and may show a mild mosaic-like chlorosis. There can be no doubt that a drastic physiological derangement takes place at this stage. The number of small dark-green leaves continues to increase, and at the same time the older affected leaves develop marginal necrosis and quickly senesce while leaves of only slightly younger age develop " pitting " and mild chlorosis.

Normal tiller elongation does not take place, and consequently the plant develops a rosette-like habit. At flowering, many of the older leaves have died, and the small dark-green leaves are particularly conspicuous. They occur for the most part on extremely short tillers close to the central axis of the plant; some, however, are found on moderately elongated tillers but are confined to the mid-region, leaves at both the base and tip of the tiller being much less reduced in size and almost normal in colour. Eventually, the small leaves show some pitting and develop marginal necrosis, but the retrogressive changes take place at a much slower rate than in the larger leaves.

Reduction in plant dry weight is, of course, more obvious in acute deficiency and includes, in addition to the two causes mentioned by Richards and Templeman (loc. cit.), the effect of reduced size of individual leaves. Not infrequently affected plants may die outright long before those normally fertilized with potassium. Almost invariably they are extremely stunted and produce only small seed yields.

(ii) *Mineral Status of Deficient Herbage.*

From evidence presented in the first part of this paper (loc. cit.) the relative amount of potassium in the tops of deficient plants was of the order of 0·7 per cent. A summary of results for leaf, and leaf + petiole samples collected in the field is given in Table 6. It will be noted that, even in the absence of external symptoms, potassium concentrations are not high: healthy leaves (and leaves + petioles) contain approximately 0·9 per cent. K. Furthermore, from other studies on Crawley sand, application of potash fertilizer seldom raises the level of potassium much beyond 1·5 per cent. during the month samples have usually been collected, viz., September.

TABLE 6.—RELATIVE AMOUNT OF POTASSIUM (PER CENT. DRY WEIGHT) IN SELECTED SAMPLES OF DWALGANUP SUBTERRANEAN CLOVER.

Date Collected	Description	Percentage K
Sept. 15, 1943 (leaf + petiole)	(a) Healthy . .	0·93
	(b) Leaves showing chlorosis and marginal necrosis	0·56
Sept. 28, 1944 (leaf only)	(a) Healthy . . .	0·93
	(b) Symptoms identical with 1943 (b) ..	0·38
Sept. 19, 1945 (leaf only)	(a) Healthy ..	0·88
	(b) Leaves showing slight bronzing and small reddish-brown stained areas (mild deficiency) .. .	0·44
	(c) Leaves very small, mostly dark-green, but sometimes with mild chlorosis (acute deficiency)	0·36

In Dwalganup subterranean clover the stem fraction is some 30 per cent. higher in potassium than the leaf (unpublished data), so that the relative amount in the tops will depend upon the nature of the analysed fraction. From the data available it seems that deficiency symptoms may be expected in Dwalganup subterranean clover when the potassium content of either leaf or leaf + petiole falls below 0·8 per cent. It is of interest that this figure agrees substantially with those reported for deficiency in apples (Burrell and Cain, 1941), citrus (Chapman and Brown, 1943), grapevines (Herschler, 1936), and lucerne (Hunter *et al.,* 1943).

Recently, Richards (1944) has stressed the relationship between diagnostic symptoms of potassium deficiency and the relative contents of other ions. He states, *inter alia,* that " it 'is a characteristic of potassium deficiency that other elements accumulate in the cells," and in reviewing evidence of other workers points out the importance of calcium and magnesium in this regard. His own work with barley clearly demonstrates the importance of sodium also, even in nutrient media high in calcium (Richards and Templeman, 1936).

In the previous paper (loc. cit.) evidence was provided that the relative amount of sodium is higher in potassium deficient than in healthy plants, in a medium relatively low in calcium (see Table 2). Even when the supply of calcium is high, the relative amount of calcium in the plant is scarcely affected, while sodium shows a small increase. Magnesium does not appear to play an important role.

Under field conditions, where phosphorus is applied as super-phosphate, no sodium is normally provided in the fertilizer. In view of the fact that calcium constitutes about 70 per cent. and magnesium 25 per cent. of the exchangeable cations in the virgin soil, it is of particular interest that a number of analyses have shown no appreciable increase in the relative amounts of either calcium or magnesium in potassium deficient plant samples. The mineral content of the 1945 sample of Table 6 was as follows:—

—	k	Ca	Mg	Na
	%	%	%	%
(a) ..	0·88	2·04	0·4	0·56
(b) ..	0·44	2·69	0·3	0·97
(c) ..	0·36	1·82	0·3	1·23

Variation in relative amount of calcium is here greater than usual and no explanation can be offered for the high (*b*) value. The sodium figures, however, show definite increase with decreasing levels of potassium, and are suggestive of a specific response to potassium deficiency in Dwalganup subterranean clover.

5. Discussion

The first factor examined by way of explanation of the differential response of subterranean clover to potassium in the years 1941 and 1942 was rainfall. The Institute records (Table 7) showed that the total quantity of rain during the growing periods was not very different —28·5 in. in 1941 and 29·5 in. in 1942. An important feature of the

1941 season, however, was that almost 18 in. fell in the two months following germination, so that leaching of potassium was to be expected.

TABLE 7.—MONTHLY RAINFALL (IN INCHES) AT INSTITUTE OF AGRICULTURE, PERTH, 1941–1945.

—	1941.	1942	1943	1944	1945.
January ..	0·09	0·09	0·95	0·17	0·05
February ..	0·07	0·08	0·49	0·03	0·07
March ..	0·22	2·53	1·01	0·17	0·59
April	3·27	2·64	2·53	1·39	0·20
May	2·24	5·92	2·33	5·10	4·75
June	9·88	9·56	6·09	3·82	13·41
July	8·06	4·13	6·07	5·85	5·06
August ..	3·98	5·08	5·89	4·16	9·25
September ..	4·18	1·45	4·04	2·59	3·48
October ..	2·07	2·41	1·06	0·58	0·61
November ..	0·86	0·30	0·24	1·56	1·96
December ..	0·08	1·12	0·00	1·27	0·91
Total ..	35·00	35·31	30·70	26·69	40·34

Results obtained from Experiment 1 (see Table 1) provide no positive evidence for the hypothesis that, in a high rainfall year, excessive leaching might eliminate differences between K and No K treatments. Admittedly, leaching may have taken place in the above trial, but the mean yield for the No K plots was moderately high—15 cwt. per acre of clover—and, in addition, no acute potassium deficiency symptoms were observed at any stage. Furthermore, the main trial extended over 1944 and 1945, two years of abnormally low and high rainfall respectively. Potassium responses were of the same order of magnitude in each season, and even though the second year results were partly dependent upon the first, the fact that rainfall was very high in the second season should, if the hypothesis were correct, have reduced the magnitude of the response to potassium. Already, evidence has been given which might account for the lack of response to potassium in Experiment 2. Leaching losses under field conditions at the Institute may, therefore, be rather less than expected on general grounds; Peech (1941), for instance, has stated that potassium is subject to rapid leaching in sandy soils, independent of pH

One observation bearing directly on the above question should be mentioned. The author has noted the striking effect of soil cultivation—more particularly, deep cultivation—on the incidence of potassium deficiency symptoms; invariably, these appear after cultivation, whilst adjacent non-cultivated areas remain almost free of symptoms. The reason is not known, but it is suggested that cultivation leads to mineralization of organic matter with release of potassium, which is subsequently subject to considerable leaching losses. In the equilibrium equations: non-exchangeable K $\overset{I}{\underset{II}{\rightleftharpoons}}$ exchangeable K $\rightleftharpoons$ K in soil solution (Peech and Bradfield, 1943), loss of organic matter would enable II to proceed to the right and

also arrest I in the same direction. The possibility cannot be over-looked that the cultivation treatments in Experiment 4 may have been responsible for revealing differences due to potassium which were not evident in Experiment 2.

A point of no small technical interest has to do with the development of symptoms in acute potassium deficiency of subterranean clover. In the older chlorotic leaves marginal scorching and premature senescence is undoubtedly associated with high succulence, although not a direct result of it. This succulence, also, is in all probability a consequence of low carbohydrate level, but the increase in relative amount of sodium may play some part as suggested by the work of Richards and Shih (1940). From work so far conducted at the Institute, it is clear that the " first " stage of potassium deficiency, as characterized by Wall (1939) for the tomato plant, viz., retardation of growth, hard type of plant, accompanied by chlorosis and mottling of the upper leaves and accumulation of carbohydrates, does not occur in subterranean clover. From Wall's tentative explanation (p. 153) it is possible that light intensity and winter temperatures at the Institute were not sufficiently low for this stage.

The later stage of deficiency in subterranean clover wherein leaf-size is greatly reduced can scarcely be assigned to potassium *per se,* and is of interest in relation to nitrogen metabolism. Even though tillering rate and leaf number are not seriously affected by potassium deficiency in subterranean clover, this reduction in leaf-size is suggestive of a direct role of potassium in nitrogen metabolism as indicated by Wall (1940) and others. It is necessary to bear in mind that the absence of effect on rate of leaf production and tillering in the above instance may be directly related to a specific effect of high sodium concentrations (see Gregory, 1937).

Of the plants so far studied—Dwalganup and Mt. Barker subterranean clover, W.A. blue lupin, Wimmera ryegrass, perennial veldt grass, and capeweed—only the clover has shown marked and significant responses to potassium. This is in general agreement with the statement of Baver (1943) that legumes respond more readily than grasses. The fact that lupins grew satisfactorily without potash fertilizer may have been due to a genetic characteristic: lupins apparently possess the ability to extract difficultly-available soil potassium (Rathsack and Laufer, 1936).

In conclusion it is pertinent to remark that no data are available with respect to two important practical field problems: (*a*) the amounts of potassium required and residual effects, and (*b*) the extent of deficiency areas in the south-west of Western Australia. The first calls for further investigation, the intensity of which will depend to some degree on (*b*). At present there is a dearth of quantitative evidence on the effects of potassium deficiency on pasture plants in southern Australia generally.

6. Acknowledgments

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Note on the Hilsch Centrifugal Jet or "Maxwellian Demon"

By R. W. Muncey, B.E.E.[*]

Summary.

From the meagre details available, a small device, used during the war in Germany for replacing ammonia pre-cooling apparatus in a liquid-air machine, has been constructed, and its operation as a possible means of maintaining reduced temperatures in cold conditioning boxes investigated semi-quantitatively. Temperatures as low as 35 deg. C. below atmospheric have been obtained under a variety of conditions examined, and there appears little doubt of the possible efficacy of the device.

During the war, R. Hilsch in Germany used a small device, since referred to as the Maxwellian Demon, to replace the usual ammonia pre-cooling apparatus in a liquid-air machine (1, 2). This device, illustrated in Figs. 1 and 2, consists of a T-shaped tubular structure with a spiral central cavity. The flow in one arm (the left in Figs. 1 and 2) is controlled by a gate valve—and in the second arm (the right) is constricted by a small centrally-placed orifice. When compressed gas is forced through the central leg of the T, the setting of the gate valve determines the relative proportions and temperatures flowing in the arms. With the valve fully opened, gas at the temperature of the input issues at the left and air is drawn in at the right. When the valve is slowly closed, a stage is reached at which a stream of cold gas flows out at the right and that issuing from the left is correspondingly warmed. Further closing of the valve causes a greater decrease in the temperature of the gas issuing at the right until a maximum effect is attained and, beyond this point, the cooling decreases until all the gas issues at the right.
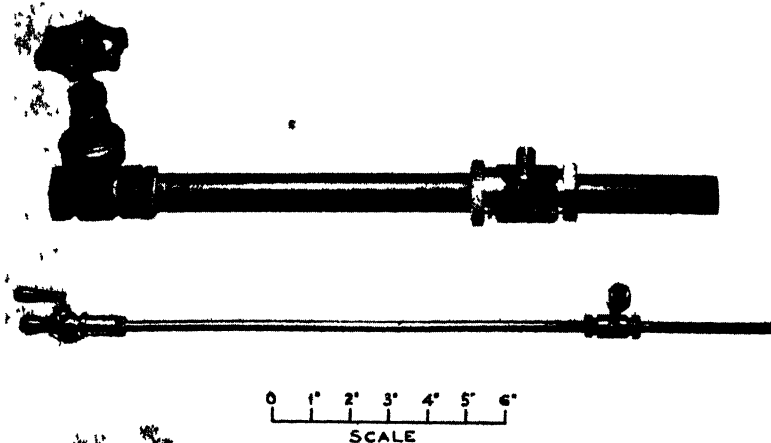
FIG. 1.—Photograph showing the relative sizes of the two Hilsch Centrifugal Jets constructed.

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Apart from the very brief description of the unit itself given in the May and December issues of the Industrial Edition of *Industrial and Engineering Chemistry* for 1946 (1, 2), no definite information on its operation or performance or on the theory behind its operation, has yet become available. However, since the problem of maintaining reduced temperatures in cold conditioning boxes is a major problem in the Building Materials Research Laboratory, it was decided that the device was worth local investigation. Based, therefore, upon the meagre details available, two units of widely differing sizes were constructed in the laboratory workshop and their undoubted success has already indicated the possibility of the device as a refrigerating agent. While the results so far to hand are admittedly only of a semi-quantitative nature, they are considered of sufficient value to pass on to others who may be interested in the utilization of the unit.

LETTERS	A	B	C	D	E	F	G	H	J
SMALL JET	3·90	4·43	·062	·250	·187	·156	·109	·062	·046
LARGE JET	11·62	5·125	·250	1·00	·890	·593	·375	·500	·187
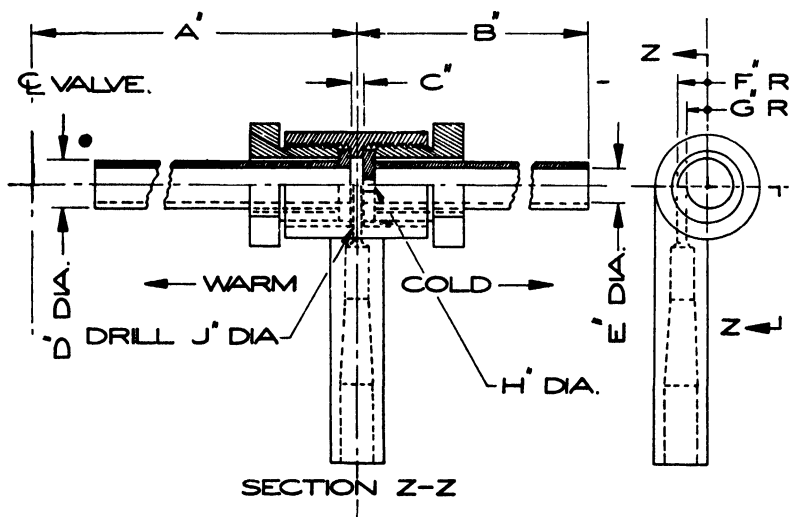
FIG. 2.—Hilsch centrifugal jet (Maxwellian Demon).

The spiral used in the unit is illustrated in Fig. 3 and is approximately of the form $r = a - b\theta^2$ (where r and θ are the polar co-ordinates of points on the curve), the constants being arranged so that a is the required maximum radius and $b(2\pi)^2$ is equal to the inlet jet diameter. This spiral is uniform in a direction along the axis of the arms of the T, and its width in that direction is slightly greater than the diameter of the jet. Its shape can readily be seen in Fig. 3. The jet is formed so that it is perpendicular to the radius vector at $\theta = 0$ and $\theta = 2\pi$ and its axis is distant $a - 2b\pi^2$ from the origin for the spiral. Other relevant dimensions of the two units constructed are shown in Fig. 2.

For the smaller unit the dimensions adopted were approximately those of the German model. This unit proved immediately successful in its trial operations. Compressed air at 90 lb. per sq. in. was used, and air noticeably colder than the surrounding atmosphere issued from the right arm of the demon when the compressed air was turned on and the gate valve adjusted roughly. Further adjustment of the valve indicated that a minimum temperature, about 35 deg. C. below atmospheric, was reached at one particular setting of the valve which eventually proved to be that at which 35 per cent. of the total air introduced was issuing through the cold jet. The maximum cooling effect, however, was reached when the volumes of air issuing at the
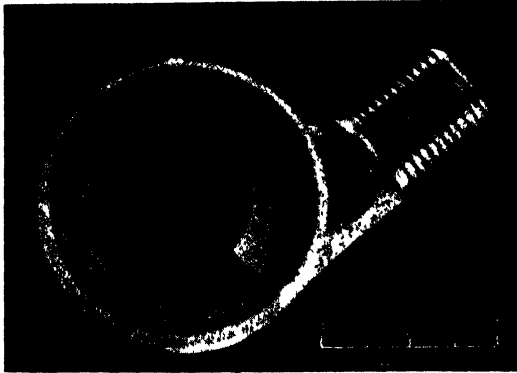
FIG. 3.—The spiral cavity of the large unit.
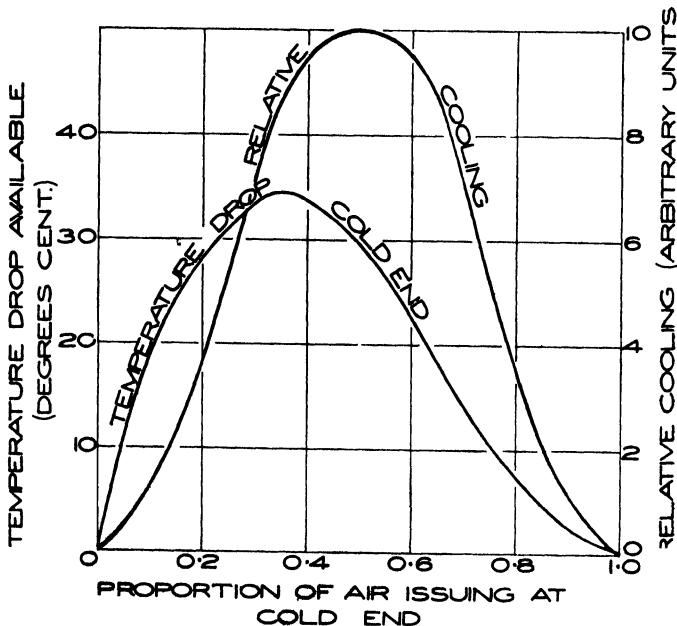
FIG. 4.—Effect of alteration of valve setting.

arms were equal and while changes in the setting of the valve necessarily resulted in corresponding alterations in the relative proportions and temperatures of air issuing from the hot and cold ends, the total flow remained reasonably constant at approximately 800 ml. per second at the pressure of 90 lb. per sq. in. The effective temperature drop in the air issuing from the cold jet, corresponding to a complete range in valve settings from open to shut, with the compressed air at 90 lb. per sq. in. is shown idealistically in Fig. 4.

Reduction of input pressure reduced the temperature drop of the cold air materially, but it was found that the setting of the valve for maximum drop remained constant. The progressive drop in temperature corresponding to compressed air inlet pressures from 0 to 100 lb. per sq. in. is illustrated in Fig. 5. No attempt has yet been made to measure the temperature drop with pressures higher than 100 lb. per sq. in., but icing up of the output tube was seen to occur far more rapidly with a pressure of about 1,000 lb. per sq. in.
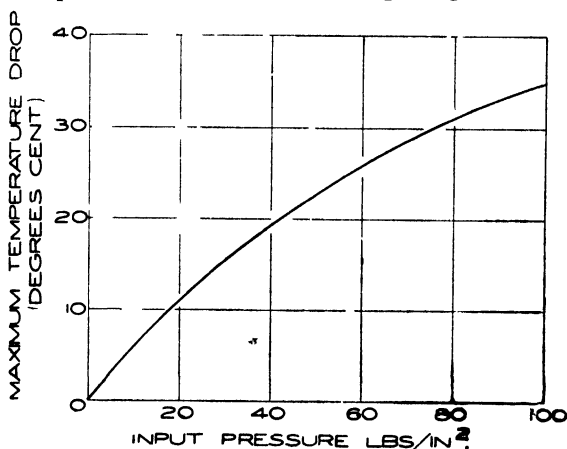
FIG. 5.—Effect of change of pressure upon temperature drop. (Optimum valve setting remained constant.)

Since for practical refrigeration purposes, much longer lengths of tubing through which to pass the cold air would be required, trials were carried out to determine the effect of change of length and of bending and coiling of the output tubes. Alteration of the lengths of both the hot and cold arms from 2 in. to 48 in. had no effect on the maximum temperature reduction of the cold air stream or on the setting of the valve. Bending or coiling of the tubes was similarly without any noticeable effect.

The second model constructed was considerably larger than the first, with the internal dimensions approximately four times those of the former; the relative sizes may be seen in Fig. 1. All tests showed that, within the limits of experimental error, the temperatures available were identical with those obtained with the smaller instrument.

From theoretical considerations in one of the articles describing the original centrifugal jet (2), it seemed likely that a reduction in the size of the cold output orifice should increase the drop in temperature of the cold air stream. Using the larger model for convenience,

the effect of varying the dimensions of the cold orifice was investigated, but it was found (as may be seen from Fig. 6) that, as the diameter of the cold outlet was either decreased or increased relative to that of the warm orifice, the temperature drop diminished in both cases. It is apparent, therefore, that the cold opening used in the original Hilsch jet is about the optimum.
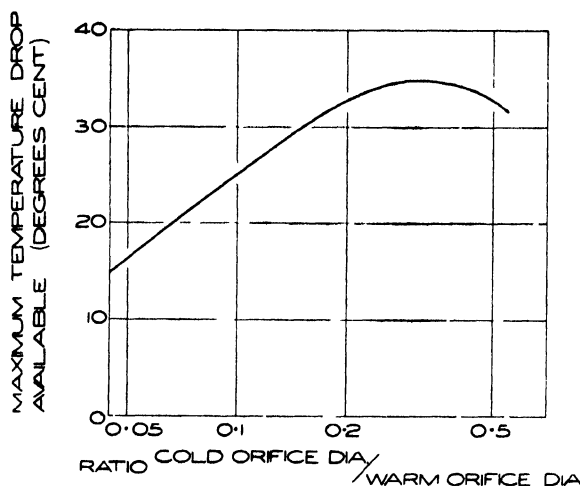
FIG. 6.—Effect of variation of cold orifice diameter on temperature drop. (Valve setting required adjustment for each new orifice diameter.)

While no suitable theory has been evolved to explain the operation of the Hilsch Centrifugal Jet or "Maxwellian Demon," the operation of the units constructed in the Building Materials Research Laboratories indicates clearly that such units show promise of being used to maintain temperatures below atmospheric, in constant-temperature boxes. For design purposes, it is clear that proportional alterations in the size of the jet units and increases in the lengths of the arms will not affect the results obtainable, and it is now proposed to make a number of these jets for use on a practical scale in these laboratories.

References

1. Anon. (1946).—*Ind. Eng. Chem.*, Ind. Ed. (Advert. Sect.) 38: 5 (May).
2. Anon. (1946).—Ibid. (Advert. Sect.) 38: 5 (Dec.).
NOTE.—Since the writing of the above article, a further reference has been noted, viz., R. Hilsch (1947).—*Rev. Sci. Instrum.* 18: 108 (Feb.).

Note on Probable Ionization by the Giacobinid Meteor Shower

By G. de V. Gipps, B.Sc.

Summary.

A description is given of the effect of the Giacobinid meteor shower of October 10, 1946, on the ionosphere as shown on normal ionospheric h',f records. The possibility of meteorites being responsible for ordinary Es ionization is considered.

No special measures had been taken to observe possible ionization by this meteor shower on October 10, 1946, but subsequent examination of the h',f records of the ionospheric recorder showed ionization below the normal E-layer at that time.

The occurrence of Es at Brisbane is a minimum in September and relatively little occurs during the early part of October, thus any ionization caused by a meteor shower probably would not be complicated by the presence of ordinary Es ionization. The maximum of the shower is reported to have occurred at about 1345 hours, 150 East meridian time (1, 2) and consequently would not be seen visually. Two examples of the records obtained are given: Plate 4, Fig. 1, is the record at 1300 hours on which the first indication (isolated spots) of unusual ionization is visible. From 1330 to 1430 hours the echoes were heavy and continuous as shown in Plate 4, Fig. 2, at 1430 hours. After this the effects became weaker and finally disappeared at about 1630 hours.

The equipment used is an automatic h',f recorder with a frequency sweep of $2 \cdot 2$ to $12 \cdot 5$ Mc/s. in $2\frac{1}{2}$ minutes. The peak power output is about 200 watts and the radiation pattern of the "Berkner" (dipole) aerials varies with frequency. Every sixth sweep is recorded photographically.

From examination of the film the chief characteristics were—

(1) The abnormal echoes were generally weak.

(2) There was no indication of blanketing, i.e. the ionization was transparent and the normal ionospheric layers were seen through it.

(3) With few exceptions the echo heights were well defined: echoes from three different apparent heights were resolved in one case (1330 hours) and preserved their identity for at least $1\frac{1}{2}$ minutes.

(4) Before and after the maximum, isolated echoes were recorded, possibly corresponding to single meteors.

(5) There was no marked change in absorption during the period of the shower.

(6) Examination of records for preceding and following days did not reveal any similar ionization, although occasional transient echoes were recorded.

* A member of the staff of the Physics Department of the University of Queensland, working in co-operation with the Council's Radio Research Board.

From the characteristics of the echoes received, it is concluded that the radio waves were returned by a process of scattering from an area of low but irregular ionization density. If a high ionization density had occurred below the *E*-region the absorption of low frequency radio waves would have increased very considerably. The records are not suitable for accurate height measurement but by using all available information from the film the strongest echoes seem to have been in the range 100 to 110 km. Some echoes had apparent heights up to 180 km. but these would probably be oblique ranges.

Since it took an abnormally intense shower of meteors to produce a weak ionization it is hardly likely that meteors play an important part in the direct production of *Es* though they may supply material (e.g. sodium) which under the ionizing influence of the sun affects the *D*-region absorption (3).

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Acknowledgment

References

1. Hey, J. S. (1946).—The Giacobinid meteor shower   *Nature* 159 (4030) 119.

2. Pierce, J. A. (1947).—Ionization by meteoric bombardment *Phys Rev* 71 (2): 88.

3. Passy, A., and Passy, M. E. (1942).—The temperature of the upper atmosphere. *J. Phys. et Radium* 3 8.

Mineral Chlorination Studies

4. The Beneficiation of Australian Graphite by Treatment with Chlorine at High Temperatures

*By F. K. McTaggart, M.Sc.**

Summary.

Graphite schist from the deposits at Uley and Koppio, South Australia, contains widely varying amounts of carbon which may be concentrated by flotation followed by leaching with dilute mineral acid to approximately 90 per cent. carbon. The removal of the residual mineral contaminants from such concentrates has hitherto presented a difficult problem solved only by the use of hydrofluoric acid. This paper describes the chlorination of a flotation concentrate in a tube furnace at temperatures between 1250° and 1450°C., resulting in a product having an ash content of only 0·05 per cent. The influence of temperature, chlorine rate, addition of inert gas, and the effects of carbon tetrachloride were determined.

Graphite having an ash content of 0·5 per cent. was produced using 1 lb of chlorine per pound of graphite recovered, while with the use of 2 lb. chlorine per pound, graphite containing 0·05 per cent. or less ash was produced.

1. Introduction

The graphite schist that occurs in the deposits at Uley and Koppio, South Australia, contains widely varying amounts of carbon, together with silica, ferric and aluminium oxides, calcium carbonate, &c. The samples supplied for use in this work contained 15–20 per cent. carbon. Investigational work on suitable ore-dressing methods for such ores has been done by Blaskett and Gartrell(1), Dunkin and Hart(2), and Plante(3) who have shown that concentrates containing 80–90 per cent. of carbon together with 1–5 per cent. of calcite may be obtained after several flotations provided preliminary grinding is carried far enough to liberate associated gangue. Croft(6) has shown that these, and also the lower grades of flotation concentrates (60–70 per cent. carbon) which tend to be produced when efforts are made to preserve the coarse flakes of the ore, can be freed of all gangue except silica by means of a reducing roast following by digestion with dilute sulphuric or hydrochloric acid. Thus graphite can be obtained either by flotation alone or by flotation followed by acid digestion, containing not more than 10–15 per cent. of gangue minerals most of which is either quartz or finely divided silica resulting from the decomposition of silicates. Leaching with hydrofluoric acid has, to date, proved the only effective method for removing this silica and so producing a high-grade graphite.

It is well known that chlorine attacks silica in the presence of carbon at high temperatures giving silicon tetrachloride. Under such conditions compounds of iron and aluminium are also attacked

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* An officer of the Division of Industrial Chemistry.

readily, yielding volatile chlorides. There appeared no reason why this method should not be used for the beneficiation of graphite containing impurities such as these, the chief difficulty being in connexion with the use of chlorine at 1200°C. or higher and the finding of a material suitable for the fabrication of a furnace and capable of withstanding chlorine at such temperatures.

The reaction between silica and chlorine in the presence of carbon is as follows:—

$$SiO_2 + 2C + 2Cl_2 \rightarrow SiCl_4 + 2CO$$

wt.ratios: $60 + 24 + 142 \rightarrow 170 + 56$
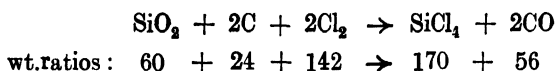
There will be some loss of carbon from the graphite owing to the formation of carbon monoxide. The magnitude of this loss will depend on the quantity of impurity to be eliminated, and in the case of a flotation concentrate such as has been described above should not amount to more than 5 per cent. There is no need for a discussion of the thermodynamics of the reaction because of the small amount of silica in the graphite. It was realized from the data available in the literature(4) that temperatures above 1200°C. would be necessary for a rapid removal of the crystalline silica, and provided the amount of chlorine used was not so great as to be excessively costly, the efficiency based on the chlorine would not need to be high.

It was, therefore, decided to determine—

    (i) the optimum temperature for the reaction,

    (ii) the optimum rate of introduction of chlorine,

    (iii) the effect of an inert gas mixed with the chlorine for sweeping out of reaction products,

    (iv) the effect of substitution of carbon tetrachloride for a certain amount of the chlorine,

    (v) the quantity of chlorine necessary to reduce the mineral impurity to approximately $0 \cdot 5$ per cent. and thereafter to approximately $0 \cdot 05$ per cent., and finally

    (vi) to find a suitable material for the construction of the furnace and to develop a satisfactory technique for carrying out the reaction.

2. Raw Materials

In early exploratory experimental work a sample of Uley graphite concentrate leached free from calcite and containing 30 per cent. ash was used. This was chosen in order to determine the general characteristics of the reaction and the likelihood of removing fairly large quantities of contaminants. This was placed in a large vertical silica tube furnace and chlorinated at 800–900°C. for two hours. The oxides of iron and aluminium were rapidly attacked and were almost completely eliminated in that time. The residual graphite contained $15 \cdot 8$ per cent. ash of a light-grey colour consisting almost wholly of silica.

For the later work a further sample of Uley flotation concentrate containing 12·2 per cent. ash was used. It occurred in flake form and had the following screen analysis—

					Per cent.
(a)	+ 48 mesh	..	..	..	38·4
	+100 mesh	..	..	..	42·6
	+150 mesh	..	..	..	10·0
	+200 mesh	..	..	..	5·0
	—200 mesh	..	..	..	4·0
					100·0

A quantity of this material was ground in a jar mill to the following size:—

					Per cent.
(b)	+ 48 mesh	..	..	..	0·2
	+ 65 mesh	..	..	..	2·5
	+ 100 mesh	..	..	..	8·9
	+ 150 mesh	..	..	..	17·3
	+ 200 mesh	..	..	..	18·8
	— 200 mesh	..	..	..	52·3
					100·0

and a further quantity was ground in the same manner to 85 per cent. minus 200 mesh (c). These three samples were leached in 5 per cent. hydrochloric acid to remove calcite and were then filtered, washed, and dried. The ash content in each case was 8·9 per cent., comprising 6·4 per cent. SiO_2 and 2·5 per cent. R_2O_3.

3. Experimental

(i) Using Platinum-wound Furnace.

In the exploratory experiments the graphite charge was heated in the vertical alundum tube furnace shown in Fig. 1A. This furnace was wound with 26-gauge " platinum — 20 per cent. rhodium " wire and insulated by means of a layer of aluminium oxide and kieselguhr bricks. The various holders used for the charge are shown in Fig. 1A. B, and C. The first three chlorinations, Nos. 4, 5, and 7 in Table 1, were carried out in the silica tube shown inside the furnace in Fig. 1A. The charge was placed in the upper section and the height of the tube was adjusted so that the charge occupied the hottest zone of the furnace. The tube was then luted at the bottom. Chlorine was passed up the centre tube into the charge and nitrogen was passed in between the tube and the furnace walls. The nitrogen served to sweep the excess chlorine and the fumes away at the top of the charge. The wall temperature was recorded by means of a platinum-rhodium thermocouple placed between the silica tube and the furnace wall and readings so obtained were correlated with the temperature at the centre of the charge by means of previous comparisons made with a thermocouple inserted in a charge, without chlorine passing. Forty grams of graphite was found to fill the tube conveniently. It was clear from these three experiments that a temperature of at

least 1250°C. was necessary to bring about a rapid removal of ash. 1300°C. appeared to be desirable because in No. 7 a sample of graphite selected from the centre of the tube was found to have an ash content of 0·1 per cent. The mean ash content of the graphite from this experiment was, however, 0·7 per cent., due almost entirely to silica flakes from the walls of the containing tube. It was obvious, therefore, that silica was unsuitable for the purpose, and attention was turned to graphite. A piece of Acheson graphite electrode (approx. 1·0 per cent. ash) was turned to the shape shown in Fig. 1B. This replaced the silica tube of Fig. 2A. Results from it were disappointing. A typical experiment, No. 8, is shown in the table. The relatively high ash content after prolonged chlorination was due to the rapid conduction of heat away from the charge by the massive lower section of the holder which projected from the bottom of the furnace. It is doubtful whether the temperature of the charge exceeded 1100°C.

(A)   (B)   (C)
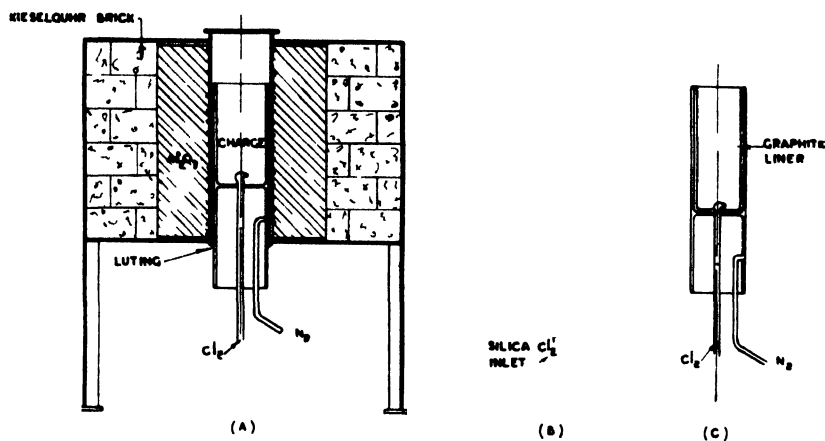
PLATIMUM WOUND FURNACE WITH   GRAPHITE   SILICA CHLORINATION
CHLORINATION TUBE   CHLORINATION   TUBE WITH GRAPHITE
TUBE   LINER

FIG. 1

TABLE 1

Experiment No	Graphite Contained in—	Charge		Temperature		Chlorine Used	Time	Residue Ash	Remarks
		Wt	Ash	Wall	Charge				
		g	%	°C	°C.	g	hrs.	%	
4	Silica tube	40	15 8		1,250		8	1 7	
5	Silica tube	40	15 8		1,200		8	8 1	
7	Silica tube	35	15 8		1,320		10	0 7	Sample from centre = 0 1% ash
8	Graphite tube	39	15 8	1,350	1,100	180	19	3	Temp. too low
9	Graphite liner	27	15 8	1,350	1,250	210	11 5	0 15	
10	Graphite liner	30	15 8	1,350	1,250	200	12	0 12	

To overcome this difficulty a return was made to the silica tube, this time fitted with the graphite liner as shown in Fig. 1c. The liner was 3/32 in. thick and had little effect on the temperature of the charge, but owing to the volume occupied by it the charge had to be reduced to about 30 grams. The results of the chlorinations (experiments Nos. 9 and 10) were encouraging. The ash content dropped to 0·15 and 0·12 per cent. respectively and 88 per cent. of the graphite was recovered in each case. However, the amount of chlorine used was excessive, being seven times the weight of the charge. Also, as in previous experiments, a white incrustation of silica collected on top of the charge and in removing this portion of the graphite was lost. The formation of this silica, which is of extreme fineness, appears to be due to hydrolysis of silicon tetrachloride immediately the vapours cease to be in contact with carbon. The hydrolysis must in turn be due to the presence of water vapour not decomposed in contact with carbon at this high temperature. The phenomenon is in line with past experience in chlorination work(5) and, in this case, the water vapour must arise from water absorbed on the graphite and gradually liberated under the action of the chlorine.

This preliminary work showed that—(a) it was possible to reduce the ash in the graphite to a satisfactory figure, and, (b) that a temperature of 1250°C. or higher was necessary for rapid decomposition of the ash. The disadvantages of the apparatus were:— (a) the chlorine was measured by flowmeter which was not sufficiently accurate to yield reliable data for chlorine consumption; (b) although diluted with nitrogen, the chlorine attacked the alundum furnace; (c) the charge was small; (d) part of the charge lifted and slid between the containing tube and the furnace walls when the chlorine rate was high; (e) there was a considerable temperature gradient between the heating element and the charge due to firstly, the alundum tube, and, secondly, the silica tube. For this reason a maximum temperature (centre of hot zone) of only 1320°C. could be attained. At this temperature the heating element was dangerously hot. It was decided, therefore, to set up a furnace that would overcome these disadvantages and yield reliable data on the factors influencing the reaction.

(ii) *Graphite Tube Furnace.*

(a) *Apparatus* (see Fig. 2).—The furnace consisted of a tube (A) turned from a length of Acheson graphite electrode (1·0 per cent. of ash). The dimensions were—

Maximum outside diameter	.. ..	4 in.
Minimum outside diameter	.. ..	2¾ in.
Inside diameter	.. ..	2 in.
Total length	.. ...	30 in.
Length of heating section .. ·	.. ..	12 in.
Length of enlarged electrodes	.. ..	7 in.

The tube was held vertically in a steel box (B) insulated by means of 200 mesh charcoal powder (C) and a double layer of kieselguhr bricks (D). Electrical connexions were made at the ends by means of water-cooled clamping electrodes (E). Bus bars (F) with flexible sections (G) served to connect the furnace to a transformer giving a

maximum of 10 volts at 2,500 amps. The furnace was provided with
two alundum inspection tubes (H) through which the maximum
temperature, i.e., at the mid point, and the temperature at the top
of the charge, could be continuously checked during operation. Also
provided was an inlet port (I) through which nitrogen was passed
during operation of the furnace. The graphite tube was tapped at
the lower end and fitted with a screw-in chlorine inlet tube (J).
From (J) a ¼-in. hole extended to the working-in section of the
furnace. At the top was a hood (K) fitted with a glass inspection
window (L) and vent holes (M). The hood was connected to an
exhaust fan. The charge (N) rested on a bed of charcoal of
⅜-in. mesh (O), and extended to the position marked (X). The
graphite sleeve (P) (see later) came to within 1 inch of the level (X).
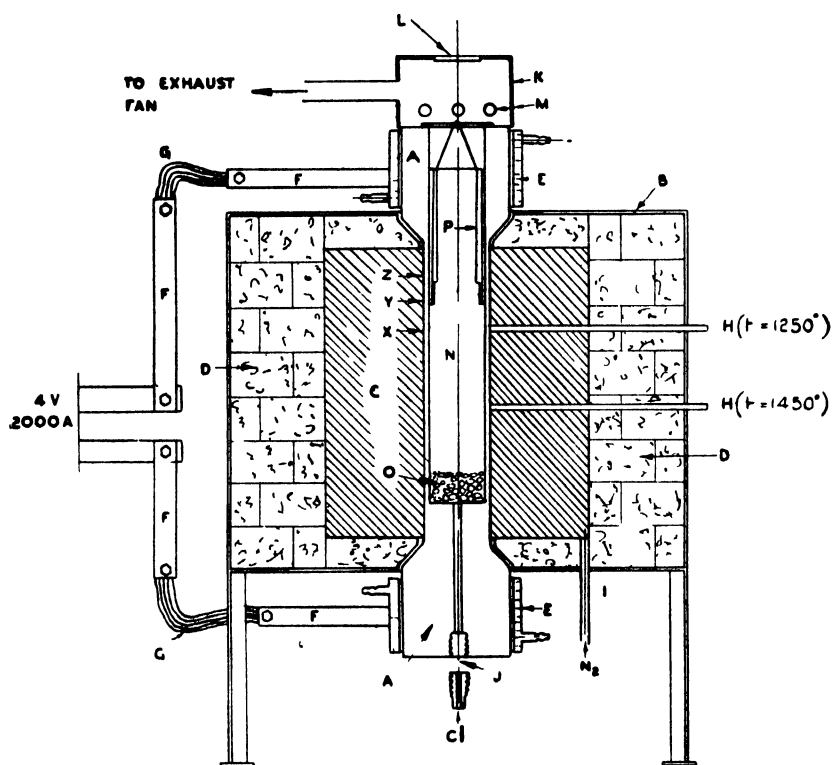
FIG. 2.- Graphite Tube Laboratory Furnace

The chlorine was supplied from a small cyclinder, 2 inches internal
diameter and 12 inches long, made from a length of steel pipe, welded
at the bottom and closed at the top with a heavy steel screw-in plug
to which was fitted a brass needle valve. This cylinder, which held
about 240 g. of chlorine and weighed when full 3·1 kilograms, was
filled from a commercial chlorine cylinder by immersing the former
in an' alcohol–dry ice mixture and allowing chlorine to distil over.
Chlorine consumption was determined by weighing the cylinder on
Dayton ratio scales accurate to somewhat better than 1 gram. Rates

of chlorine flow were determined roughly by flowmeter and accurately by weighing the cylinder every quarter or half hour during an experiment.

The charge was placed in the furnace with the aid of a funnel and removed by suction into a small bag house. The bag could be weighed accurately for the determination of recovery.

(b) *Calibration of furnace for temperature.*—The furnace was calibrated by means of a platinum-rhodium thermocouple in a thin-walled alundum sheath, inserted down the centre of the tube with a charge of graphite in place. At a mid-point temperature of 1450°C. the temperature at points $3\frac{1}{2}$ inches below and $3\frac{1}{2}$ inches above was 1250°C. This zone was therefore chosen as the working region. The setting of the voltage controls and the voltage across the transformer terminals were noted, and also the temperatures as determined by means of an optical pyrometer through the observation tubes in the side of the furnace. In this way the temperature inside the furnace during an experiment could be estimated. For a maximum temperature of 1450°C. the potential across the furnace was 3·85 volts and the current 1950 amps. In the 7-in. working zone a 70 gram charge could be accommodated.

Initially the charges were chlorinated without the graphite sleeve shown in Fig. 2, but it was found that a layer of oxides formed on the inside wall of the furnace immediately above the top of the charge. These oxides, produced apparently by the reaction discussed previously, then reacted with the graphite wall, resulting in severe pitting. As before, a considerable amount of silica collected in the top layer of the charge and this was removed by suction before the bulk of the graphite was taken out. To prevent pitting of the walls and in order to obviate the waste of the top $\frac{1}{2}$—1 in. of the charge, several experiments were made using a " iopping " of graphite up to the level indicated by (Z). This effectively stopped the pitting but caused difficulty in filling and emptying, because of the tendency for mixing to take place between the charge and the " topping." The problem was finally solved by the use of the graphite sleeve shown in position in Fig. 2. The upper section of this liner was 3/16 in. thick and the lower $2\frac{1}{2}$ in. was 1/16 in. thick. Its effect on the temperature distribution was negligible and it was necessary to place only an additional $1\frac{1}{4}$ in. of graphite on top of the charge. This layer was removed with the silica after chlorination. The sleeve was slowly pitted owing to deposition on it of oxides and was replaced from time to time as required.

(c) *Influence of chlorine rate.*—In the experimental work described in this section and in (d) and (e) below, graphite of the type C, i.e., 85 per cent. minus 200 mesh, was used. It was recognized that from the point of view of heating and other operating costs, in an eventual industrial application of the process, the shorter the time of chlorination the better. Hence several experiments were conducted to determine the optimum rate at which chlorine should be passed. The maximum rate practicable was found to be 30g./hr., as at rates faster than this the graphite was carried up the tube by the gas stream. The results cited in Table 2 show that this maximum rate was most effective in bringing about the reduction of the ash. At 30 g./hr. a reduction of ash content from 9 per cent. to approximately

0·5 per cent. was achieved in 2·3 hours, i.e., with a consumption of 70 g. of chlorine (experiments Nos. 19 and 20). When 10 g./hr. of chlorine was passed for 7 hours, again a consumption of 70 g. chlorine, the ash fell to only 4·6 per cent. (experiments Nos. 13 and 14), while at 20 g./hr. an intermediate figure was obtained (experiment No. 18).

TABLE 2.

Experiment No.	Charge Wt.	Chlorinating Agent.		Time	Ash.		Cl₂ Rate	Cl₂ Content of Graphite.	Remarks
		Type.	Amount.		Initial.	Final.			
	g.		g.	hrs.	%	%	g./hr.	%	
15	70	Cl₂	70	7	8 9	4 6	10	..	
16	70	Cl₂+ CCl₄	70 + 26	7	8 9	4 7	10	..	CCl₄ added
18	70	Cl₂	70	3 5	8 9	2 1	20	..	
19	70	Cl₂	70	2 3	8 9	0 59	30	0 7	Not gas washed
20	70	Cl₂	70	2 3	8 9	0 43	30	0 19	Gas washed with N₂
21	70	Cl₂	70	2 3	43	0 02	30	..	
23	70	Cl₂	70	2 3	59	0 05	30	..	
24	70	Cl₂	34	3 4	8 9	6 1	10	..	N₂ sweep 2 cc./min.
25	70	Cl₂	39	2 6	8 9	2 25	15	..	N₂ sweep 78 cc./min.
27	70	Cl₂ + CCl₄	47 + 23	2 3	8 9	0 58	20	..	CCl₄ substituted
29	200	Cl₂	200	6 6	8 9	3 37	30	..	Flake graphite
30	200	Cl₂	200	6 6	8 9	3 34	30	..	Flake graphite
31	110	Cl₂	110	3 6	8 9	2 71	30	..	52°₀, — 200

(d) *Influence of inert sweeping gas.*—In the elimination of approximately 9 per cent. ash, which corresponds to 6·3 g. silica, not more than 15 g. chlorine would be used, the remaining 55 g. merely serving to sweep away the reaction products. The greater efficacy of the fast chlorine rate appeared to be connected with this rapid removal of products but later it was found to be dependent on another factor also. Graphite containing approximately 0·5 per cent. ash, the product of experiments Nos. 19 and 20, was treated again in the furnace at 30 g./hr. chlorine for 2·3 hours. In one case, No. 21, the ash fell to 0·02 per cent., while in the other, No. 23, it fell to 0·05 per cent. In all these cases the recovery of graphite was better than 90 per cent., based on the graphite (carbon) content of the original sample taken.

Because the greater part of the chlorine appeared to be effective only in sweeping out the reaction products, it was decided to investigate the effect of the substitution of an inert gas such as nitrogen for some of the chlorine. Accordingly experiments were carried out using chlorine 10 g./hr. and equal volume of nitrogen for 3·5 hrs. (Expt. No. 21); and chlorine 15 g./hr. and equal volume of nitrogen for 2·3 hrs. (Expt. No. 25). The results are in no way comparable to the chlorinations in which the whole volume of the gas was chlorine.

This must be due to the fact that the concentration of the chlorine at the graphite-silica surface is reduced by the admixture of nitrogen. The reaction rate thus falls off.

However, the results using chlorine were considered satisfactory. Graphite containing 0·5 per cent. ash could be produced from flotation concentrate by the additional expenditure for chlorine of approximately 4d. per pound of graphite produced plus other operating costs. For a further 4d. per pound a high grade of graphite containing only 0·05–0·02 per cent. ash could be produced. Accurate costing could only be done after pilot plant or large-scale experimental work, but it appears certain that graphites could be produced as cheaply as imported grades.

(*e*) *Influence of carbon tetrachloride on the chlorination.*—Several experiments were carried out in order to determine the effect on the reaction rate of the addition of carbon tetrachloride. When carbon tetrachloride was passed in large amounts the carbon tetrachloride decomposed and carbon was deposited as a hard tube. Through it the chlorine and carbon tetrachloride were carried up into the middle of the charge and the lower regions were found to contain a relatively large ash. When a portion only of the chlorine was replaced by this reagent as in Expt. No. 27, no carbon tube formed but the results were not significantly different to those obtained from corresponding experiments in which no carbon tetrachloride was present.

(*f*) *Chlorination of coarser grades of graphite.*—The lamellar structure of graphite suggested that it might be possible to chlorinate the leached concentrate without recourse to fine grinding. However, the experimental data found in Expts. Nos. 29, 30, and 31 showed that, like other minerals studied to date, the graphite must be ground finely for efficient chlorination. The flake graphite (sample A) showed a drop in ash content from 8·9 to 3·35 per cent. (mean of Expts. Nos. 29 and 30) under conditions that brought the ash of the standard graphite (C) to 0·4–0·6 per cent. Graphite B chlorinated more readily and under similar conditions the ash dropped to 1·74 per cent. but the merit of this result is difficult to assess without reliable data on the cost of grinding. It may well be that for some purposes chlorination of graphite of this size would be preferable.

(iii) *Adsorption of Chlorine on Graphite.*

For many purposes such as lubrication, graphite must be free from materials that will cause abrasion, or give rise to corrosion, even under moist conditions such as sometimes obtain in bearings. Samples of graphite from Expt. No. 19, which had been chlorinated to 0·5 per cent. ash and allowed to cool in an atmosphere of chlorine, were analysed for chlorine by the combustion method and found to contain 0·7 per cent. chlorine. If this figure arose from free chlorine the graphite would be quite unsuitable for lubrication. Hence further samples were agitated with 1N sodium hydroxide and with distilled water for 24 hrs., after which the graphite was filtered off and the filtrates tested for chloride with silver nitrate. In each case only the slightest cloudiness was apparent. No attempt was made to estimate the chloride liberated as nephelometric apparatus was not available. At the conclusion of Expt. No. 20 the chlorine was turned off and

nitrogen was passed through the charge at about 50 cc./min. for 0·5 hr. before the temperature was allowed to fall, and thereafter until the charge was cool (about 2 hrs.). The combustion method of analysis showed that there was 0·19 per cent. chlorine present but no trace of chloride could be detected by the other analyses. The chlorine was apparently so firmly bound to the graphite that even prolonged treatment with alkali would not remove it. Croft(6) has thoroughly investigated this property of graphite and the above results are in agreement with his work. It appears, therefore, that graphite prepared by chlorination and gas washed as described would be unlikely to give rise to corrosive compounds under normal conditions.

4. Acknowledgments

5. References

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5. McTaggart, F. K —*J. Coun. Sci. Ind. Res.* (Aust.) 18: 5 (1945); Ibid. 18: 424 (1945).

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NOTES

Changes of Title of Imperial Agricultural Bureaux

As a result of a recommendation of the 1946 Review Conference of the Imperial Agricultural Bureaux, the Imperial Bureau of Pastures and Forage Crops now becomes the Imperial Bureau of Pastures and Field Crops (Director: Dr. R. O. Whyte). In its expanded form it will cover that part of the literature on the following crops not already the concern of the Bureaux of Plant Breeding and Genetics, or Soil Science; all cereals, field root crops, pulses, groundnuts, cotton and other fibre crops grown on a field scale, sugar beet, and sugar cane, in addition to the literature on grassland management and fodder production which has been the concern of the Bureau for the past seventeen years. Attention will continue to be given to those aspects of plant biological research which refer to the crops now covered and sections will now also deal with farming systems concerned in the production of field or forage crops, and certain economic aspects of crop and fodder production. Although the name of the Bureau has already (as from April 1, 1947) been changed to Pastures and Field Crops, and a second abstracting journal (in addition to *Herbage Abstracts*) is to be published from 1948 onwards, it will be some time before it will be possible to claim that the extended field of research and practice is being adequately covered, or before it will be possible to reply without a little difficulty to inquiries on the new crops now added to the Bureau's responsibilities. It will be much appreciated if research workers, Institutes, and Departments concerned with these crops will send their publications and reports to the Imperial Bureau of Pastures and Field Crops, Penglais, Aberystwyth, Wales, for review in the new abstracting journal dealing with field crops. Particulars of this journal can be sent to persons interested.

Acting on the recommendation of the 1946 Conference the Executive Council of the Bureaux also decided that the Imperial Parasite Service would become the Imperial Bureau of Biological Control as from 1st April, 1947.

Review

" GESTATION PERIODS—A TABLE AND BIBLIOGRAPHY,"

compiled by J. H. Kenneth.

(Technical Communication No. 5 of the Imperial Bureau of Animal Breeding and Genetics, 1946, pp. 30. Price 3s. Obtainable from the Central Sales Branch, Imperial Agricultural Bureaux, Penglais, Aberystwyth, Wales.)

This second edition of a communication first published in 1943 is a source book of information on gestation periods of 300 species, and contains 442 references to literature. The species are arranged in alphabetical order of common names and, in respect to each, all available sources of data are quoted on the mean, maximum, and minimum durations of gestation. For domestic animals, data referring specifically to individual breeds are listed as such.

For most species of animals, in respect to which only limited data are available, indication of the mean gestation period and its range represents the best information which can be obtained. However, abundant data are available for most domestic animals so that a more precise estimate of the normal degree of variation from the mean could be calculated. Maximum and minimum values are of little use except as curiosities, especially when the possibilities of abortion and doubts as to the validity of extremely long records are taken into consideration. For information on the standard deviations of gestation periods in domestic animals, the reader may consult Animal Breeding Plans (by J. L. Lush. 1943. Iowa State Coll. Press, Ames, Iowa. 2nd Ed.).

The 300 species listed include various aquatic mammals and Australian marsupials. The data on the human are very numerous and are in two sections, including those records for which the interval from fertilization to labour is measured and those for which the interval used is from last menstruation to labour.

Recent Publications of the Council

Since the last issue of this *Journal,* the following publications of the Council have been issued:—

Bulletin No. 203.—" Agar in Australia," by E. J. Ferguson Wood, B.A., M.Sc.

This Bulletin gives an account of the agar industry in Australia during the war, and the research that led up to it; overseas work on agar is also summarized. The uses of agar, its physical and chemical properties, and methods of testing its various qualities, are discussed, together with the sources of seaweed used as the raw material, and the manufacturing processes employed. The possibilities of the agar industry in Australia are outlined. At the present time the industry produces some 160,000 lb. of agar a year, valued at about £150,000, and it is expanding to the limits of the known beds of the particular type of seaweed required. Much of the agar produced is exported to Britain, the Argentine, and Uruguay.

Bulletin No. 204.—" A Soil Survey of Part of Waterhouse Estate, County of Dorset, North-East Coast, Tasmania," by G. D. Hubble, B.Agr.Sc.

This survey covers an area of 50,000 acres in the Waterhouse Estate lying in the coastal belt north of Scottsdale, north-eastern Tasmania. It was undertaken at the request of the Tasmanian Post-War Land Settlement Committee to examine the soils and estimate the quality of the north-eastern coastal belt for future settlement. The area has been used as sheep and cattle run country for nearly 100 years, but little cultivation has been undertaken and the stock are almost entirely dependent on the natural vegetation.

The soils are of very low fertility for the most part, and on present knowledge it seems unlikely that good pastures could be established economically on them, except in small areas. A limited subdivision of the property into holdings of from 8,000–10,000 acres, each with an initial carrying capacity of about 1,500 sheep, might be possible;

but before any settlement is considered it must be demonstrated that profitable sown pastures can be established. Field trials are being undertaken by the Department of Agriculture for this purpose. The establishment of windbreaks, the prevention of sand drift from the coast, and the provision of adequate drainage, are other problems that must be tackled.

Bulletin No. 207.—" The Fumigation of Wheat in Bag Stacks," by Frank Wilson and F. J. Gay, B.Sc., D.I.C.

Prior to the investigations described in this Bulletin, the effective fumigation of commercial stacks of bagged wheat had never been accomplished, and was generally considered to be impossible of attainment in an economic manner. The stacks to be treated were commonly 100 feet long, 70 feet wide, and about 20 tiers high, and covered by a permanent roof, so the provision of an airtight covering was virtually impossible. Experiments showed that if the stack was built on an airtight base, and airtight curtains were erected round the sides, fumigation could be effectively carried out by applying certain heavier-than-air fumigants over the surface of the stack. Carbon bisulphide at the rate of 1 lb., or methyl bromide at the rate of 2 lb. per 1,000 cubic feet gave a good kill of weevils and mice. The fumigant is not so effective in the top few tiers of wheat, but infestation is largely restricted to the lower part of the stack. This fumigation method, using carbon bisulphide, has been employed extensively in South Australia since May, 1944, and some 4,000,000 bushels of wheat have been treated with entirely satisfactory results.

Bulletin No. 209.—" Interaction of Insect Infestation, Temperature, and Moisture Content in Bulk-Depot Wheat," by Frank Wilson.

This Bulletin describes an experiment in which the changes in insect-densities, temperatures, and wheat moisture contents occurring in bulk-depot wheat inoculated with *Rhizopertha dominica* were studied over a period of ten months. This insect and *Latheticus oryzae* penetrated and bred throughout the zone studied (a depth of 5 feet) during the earlier part of the experiment, but subsequently became restricted increasingly to a shallow surface zone, owing to the development of lethal physical conditions in the wheat. Reproduction near the wheat surface was made possible by lower temperatures and higher moisture contents. Near the surface the loss in wheat weight was as much as 40 per cent., but the percentage rapidly decreased with depth and below a level of about 2 feet the damage was negligible.

Bulletin No. 211.—" The Water Retting of Flax. Résumé of Investigations from 1940 to 1945," by W. L. Greenhill, M.E., and Jean F. Couchman, B.Sc.

The methods now generally adopted for water retting flax are based on a large amount of empirical knowledge in the assimilation and use of which the Belgians have in the past undoubtedly been pre-eminent. The problem as it affected this country has been to adapt Belgian methods to local conditions and straw types and, if possible, improve on them by making retting more of a science than an art. Investigations have been conducted in water retting research both in the laboratory and at commercial plants to determine the effect of varying such factors as the temperature and duration of the

preliminary rinse, the temperature, straw to water ratio, dilution and duration of the ret proper, and the use of a final wash. Single and double retting and special retting techniques, such as the treatment of crated straw in channels, have been tried, and also the influence of such factors as the use of saline water, supplies of which occur naturally in certain of the flax districts. Precise methods for determining rate of progress and the end-point of a ret have been considered and a technique developed which, based on the buffer capacity of the retting liquor, has greatly assisted in making comparisons between different retting schedules.

As the outcome of this work, it has been possible to recommend to flax mills a basically sound retting procedure, which is economical of time, labour, water, and fuel, and can be used with confidence for the type of straw grown in this country.

Bulletin No. 217.—" The Relative Importance of Live Sheep and of Carrion as Breeding Grounds for the Australian Sheep Blowfly *Lucilia cuprina,"* by D. F. Waterhouse, M.Sc.

Lucilia cuprina is what is called a primary fly, that is, it is responsible for initiating strikes in sheep. Secondary flies are attracted to struck sheep, but are of little danger to sheep in the absence of primary flies, so control measures should be directed against the latter. Past observations have indicated that strikes in living sheep are responsible for breeding the primary fly, carcasses being far less important, so this was tested in experiments described in the present Bulletin. *Lucilia cuprina* flies developed from only 6 out of 27 carcasses exposed at Canberra at intervals throughout the year, with an average of four flies per animal, whereas an average of 10,000 secondary flies developed from each carcass. On the other hand, an average of over 1,200 *L. cuprina* per strike was produced from 26 struck sheep, and generally no secondary flies at all. Thus, the destruction of carcasses is of no value in controlling primary flies.

Bulletin No. 224.—" Mechanical Composition of Soil in Relation to Field Descriptions of Texture," by T. J. Marshall, M.Agr.Sc., Ph.D.
    This Bulletin was reviewed in the last issue of this *Journal* (20: 315).

Forthcoming Publications of the Council

At the present time, the following future publications of the Council are in the press:—
    Bulletin No. 202.—" The Strain Complex and Symptom Variability of Tomato Spotted Wilt Virus," by D. O. Norris, M.Sc. (Agric.).
    Bulletin No. 206.—"Pedogenesis Following the Dissection of Lateritic Regions in Southern Australia," by C. G. Stephens, M.Sc.
    Bulletin No. 210.—" Preliminary Survey of the Natural Pastures of the New England District of New South Wales, and a General Discussion of Their Problems," by R. Roe, B.Sc. (Agric.).

Bulletin No. 212.—" The Frictional Properties of Lead-Base and Tin-Base Bearing Alloys: The Role of the Matrix and the Hard Particles," by D. Tabor, Ph.D.

Bulletin No. 213.—" Laboratory and Field Tests of Mosquito Repellents," by R. N. McCulloch, B.Sc., B.Sc.Agr., and D. F. Waterhouse, M.Sc.

Bulletin No. 214.—" The Preparation and Properties of Synthetic Cryolite," by P. Dixon, M.Sc., and T. R. Scott, M.Sc.

Bulletin No. 215.—" Studies in the Biology of the Skin and Fleece of Sheep. 4. The Hair Follicle Group and its Topographical Variations in the Skin of the Merino Fœtus," by H. B. Carter, B.V.Sc., and Margaret H. Hardy, M.Sc.

Bulletin No. 216.—" An Examination of the Peet-Grady Method for the Evaluation of Household Fly Sprays," by D. F. Waterhouse, M.Sc.

Bulletin No. 218.—" Studies of the Physiology and Toxicology of Blowflies. 12. The Toxicity or DDT as a Contact and Stomach Poison for Larvae of *Lucilia cuprina.* 13. Insectary Tests of Repellents for the Australian Sheep Blowfly," by D. F. Waterhouse, M.Sc.

Bulletin No. 219.—" Spray Tests against Adult Mosquitoes. 1. Laboratory Spray Tests with Culicine (*Culex fatigans*) Adults," by D. F. Waterhouse, M.Sc. " 2. Spray Tests with Anopheline (*Anopheles punctualatus farauti*) Adults," by D.F. Waterhouse, M.Sc., and D. O. Atherton, M.Sc.Agr.

Bulletin No. 220.—" The Preparation and Use of Harvey's Reduced Strychnine Reagent in Oceanographical Chemistry," by D. Rochford, B.Sc.

Bulletin No. 221.—" Contributions to the Study of the Cell Wall. 4. The Nature of Intercellular Adhesion in Delignified Tissue. 5. The Occurrence, Structure, and Properties of Certain Cell Wall Deformations," by A. B. Wardrop, M.Sc., and H. E. Dadswell, D.Sc.

Bulletin No. 222.—" The Chaetognatha of South Eastern Australia," by J. M. Thomson, M.Sc.

Bulletin No. 223.—" Report of Marine Borer Survey in New Guinea Waters," by A. W. Shillinglaw, B.Sc., Dip.For., and D. D. Moore, B.Sc., A.S.T.C.

Bulletin No. 225.—" Studies on the Control of Wheat Insects by Dusts. 1. Field Tests of Various Mineral Dusts against Grain Weevils," by F. J. Gay, B.Sc., D.I.C., F. N. Ratcliffe, B.A., and R. N. McCulloch, B.Sc., B.Sc.Agr. " 2. Further Tests of Various Mineral Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C. " 3. The Use of Dust Barriers for the Control of Grain Insects," by F. J. Gay, B.Sc., D.I.C. " 4. The Use of 'DDT- and 666- impregnated Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C.

Bulletin No. 226.—" An Ecological Study of the Australian Plague Locust (*Chortoicetes terminifera* Walk.) in the Bogan-Macquarie Outbreak Area, N.S.W.," by L. R. Clark, M.Sc.

Bulletin No. 227.—" Studies on Perennial Veldt Grass (*Ehrharta calycina* Sm.)," by R. C. Rossiter, B.Sc.(Agric.).

Bulletin No. 228.—" Ecological Observations on the Small Plague Grasshopper, *Austroicetes cruciata* (Sauss.), in the Trangie District, Central Western New South Wales," by L. R. Clark, M.Sc.

Bulletin No. 229.—" Studies in Cement-Aggregate Reaction. I.—Australian Aggregates and Cements," by A. R. Alderman, D.Sc., Ph.D., A. J. Gaskin, M.Sc., R. H. Jones, B.Sc., and H. E. Vivian, B.Sc.Agr. " II.—The Effect of Alkali Movement in Hardened Mortar," by H. E. Vivian, B.Sc.Agr. " III.—The Effect of Void Space on Mortar Expansion," by H. E. Vivian, B.Sc.Agr. " IV.—The Effect of Expansion of the Tensile Strength of Mortar," by H. E. Vivian, B.Sc.Agr. " V.—The Effect of Void Space on the Tensile Strength Changes of Mortar," by H. E. Vivian, B.Sc.Agr. " VI.—The Effect of Carbon Dioxide," by A. J. Gaskin, M.Sc.

Bulletin No. 230.—" The Preparation and Properties of Aluminium Fluoride," by T. R. Scott, M.Sc.

" The Commercial Timbers of Australia—Their Properties and Uses," by I. H. Boas, M.Sc.

" Handbook of Australian Pelagic Tunicates," by Harold Thompson, M.A., D.Sc.

PLATE 1

The Variation of Tensile Strength and Modulus of Elasticity of Hoop Pine Veneer with the Direction of the Grain. (See page 338.)

Fig 1 Test rig for specimens loaded at nominal angles of from 22½° to 55 to the grain.
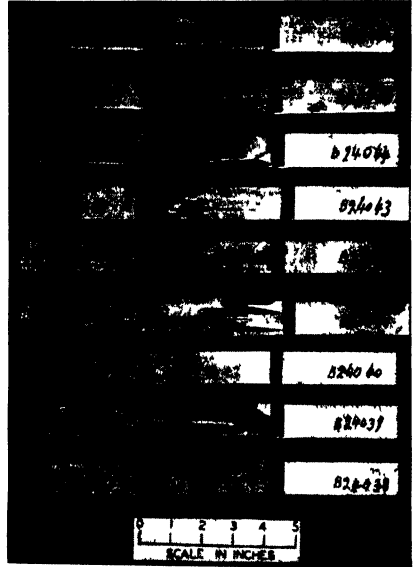
Fig 2 Typical failures with the direction of loading parallel to the grain of the veneer.
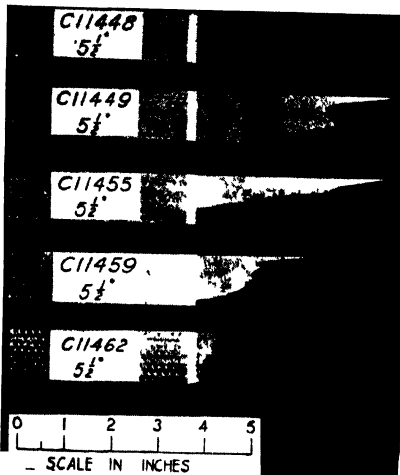
Fig 3 Typical failures with the nominal direction of loading at 5½° to the grain of the veneer
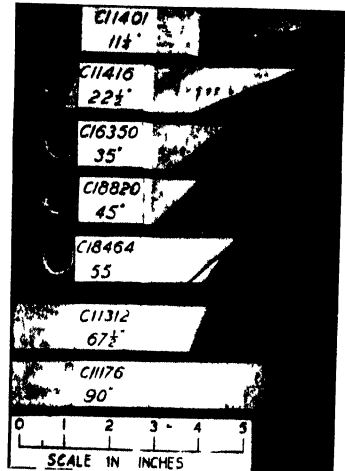
Fig 4 Typical failures with the nominal direction of loading at angles of from 11½ to 90 to the grain of the veneer

PLATE 2

Tests on Small Clear Specimens of North Queensland Kauri.
(See page 345.)
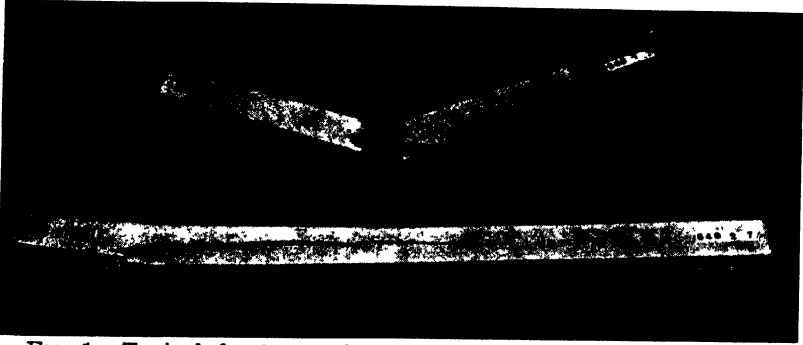
FIG. 1.—Typical fractures of centre-point and four-point bending specimens.
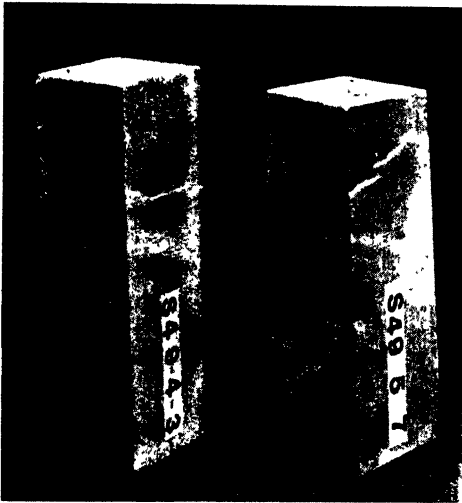
FIG. 2.—Typical failures of specimens tested in compression parallel to grain (large prism).

FIG. 3. Brittle failure of kauri under impact loads illustrated in toughness and Izod fractures

PLATE 3.

The Nature of Plastic Deformation in Wood at Elevated Temperatures
(See page 361)
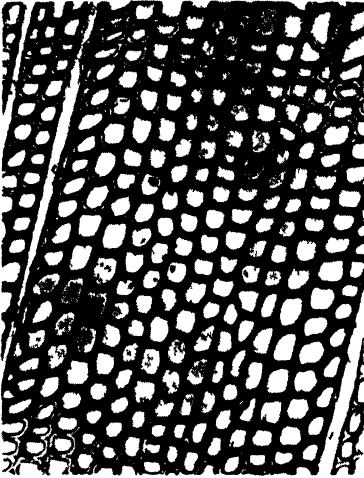
a.
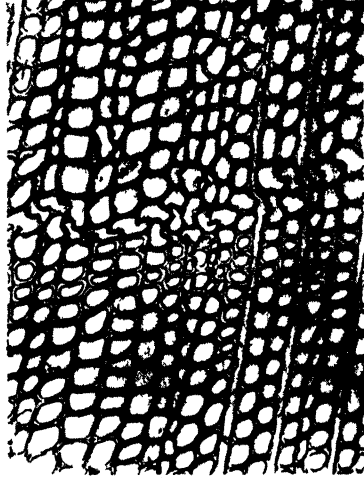
b.
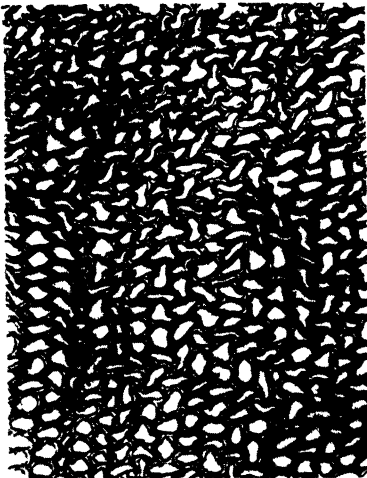
c.

d.

(a) Showing a specimen without fracture   Pressed at 100 lb sq in and 140 C

(b) Showing a specimen with incipient cell wall fracture in the early wood   Pressed at 1 000 lb sq in and 160 C

(c) Showing a specimen with advanced fracture of the cell wall   Pressed at 1 200 lb sq in and 200 C

(d) Enlargement of part of (c)

PLATE 4

Note on Probable Ionization by the Giacobinid Meteor Shower.
(See page 407.)

Fig 1   The first indications of ionization by the meteor shower occurred on records for 1300 hours on October 10, 1946   The transient echoes are at a height of about 100 km
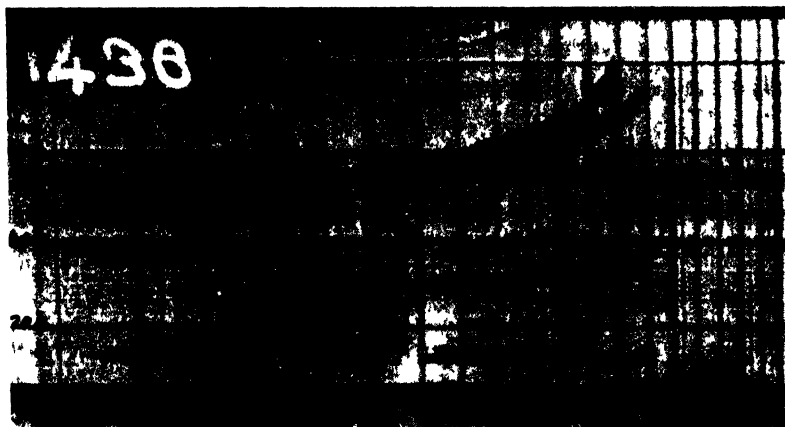
Fig. 2   At the maximum of shower the records appeared as shown, frequently with well-defined   layers" present above 100 km

The Control of Silverfish and the German Cockroach

*By T. Greaves**

Summary

Successful control of silverfish (*Ctenolepisma longicaudata* Esch.) was achieved by the application of 1½-2 lb. of 10 per cent. DDT dust in Canberra houses, each containing six to eight rooms. Using a modified hand dust-gun, 1-1½ lb. was applied to crevices, behind skirtings, architraves, cupboards, &c., and ½ lb. was applied to the upper surface of the ceilings.

The importance of the ceiling treatment was realized when failure to obtain complete control with repeated applications of 10 per cent. DDT dust in the rooms was followed by successful control when ½ lb. of the dust was applied to the upper surface of the ceilings.

For the control of silverfish infestations in offices, a pyrethrum-DDT spray is effective for treating open filing racks, &c., which are in daily use. This spray treatment is used to complement treatment with 10 per cent. DDT dust, as recommended for houses.

Re-infestation of houses from wood heaps led to a recommendation for the control of silverfish in these places, namely periodic clearing of wood debris and treatment of the lower part of the stacked wood with ¼-½ lb. of 10 per cent. DDT dust.

Successful control of severe infestations of the German cockroach (*Blatella germanica* (L.)) in large kitchens at civilian and service hostels was achieved by the application of 3-5 lb. of 10 per cent. DDT dust to crevices, especially those occurring in the warmer parts of the kitchen, such as around the stove, hot water pipes, boilers, or under sinks.

1. Introduction

Womersley (1939) records *Ctenolepisma longicaudata* Esch. as a common pest of houses and libraries in Australia, and Lindsay (1940) records this insect from as far north as Cairns, Queensland, and as far inland as Broken Hill, New South Wales. Notes from Agricultural Journals in New South Wales, Victoria, and South Australia indicate that *C. longicaudata* is a very common household pest in these States.

When large numbers of silverfish were required in 1941 for laboratory experiments at Canberra, approximately 1,000 were obtained from Brisbane. These proved to be *Ctenolepisma urbana*, a species described by Slabaugh in 1940. *C. urbana* has since been identified from Canberra, but *C. longicaudata* appears to be the predominant species in the district.

*An officer of the Division of Economic Entomology.

Silverfish are very common in Canberra. They damage artificial silk and paper of various kinds, particularly those containing starch or sizing, such as wall papers and photographs. They are especially destructive to the flimsy paper used for typing air-mail correspondence. According to Lindsay (loc. cit.) silverfish cannot live on wool or pure silk.

The German cockroach (*Blatella germanica* (L.)) is a very common pest in the kitchens of hostels and in homes equipped with a continuous hot water service. Heavy infestations developed in Service establishments erected in the Canberra district during the war years 1939-45. The Division of Economic Entomology was asked to help deal with these infestations, and it was observed that when a DDT dust was applied to the hiding places of the German cockroach, not only this pest, but silverfish in addition, were effectively controlled.

2. Previous Work on Silverfish Control

Several Australian workers have given attention, at various times, to the problem of silverfish control. G. F. Hill, in 1935, initiated the investigations carried out by the Division of Economic Entomology. Lindsay (loc. cit.) and Swan (1941) have published work on the life history, food, damage, and control measures.

Sodium fluoride and fluosilicate dusts are not satisfactory and, until the discovery of DDT, baits and sprays offered the most effective means of control. The Division of Economic Entomology (Anon. 1936, 1939) recommended barium fluosilicate incorporated in a sweetened flour paste, painted onto cards, and placed in cupboards and other places where silverfish abound. The persistent use of the C.S.I.R. bait cards reduced silverfish populations in many houses to a level at which they no longer caused important damage. Both Lindsay (loc. cit) and Swan (loc. cit.) recommended variations of the above bait.

Although barium fluosilicate bait cards have proved efficient in houses, they have been a complete failure in offices where there is a superabundance of attractive alternative food. Many thousands of cards have been distributed in Canberra offices infested with silverfish, but very few have been attacked. To cope with silverfish in Government offices, and in the Parliamentary Library, a high-grade pyrethrum spray was recommended, and a regular programme of treatment, using a spray containing 0·17 per cent. w/v pyrethrins, was initiated, each office being sprayed at approximately six-month intervals. This spray schedule effected a marked reduction in the nuisance, although control was by no means complete. Shortages of staff and materials during the war years caused a partial abandonment of this programme, and silverfish populations steadily increased and were regaining pest proportions when the Divisional investigations were resumed.

In the meantime DDT had become available, and its various uses were, being investigated. Davies (1946) and others reported promising results from preliminary tests against silverfish with DDT sprays and dusts containing 5 per cent. DDT.

3. Investigations on the Control of Silverfish in Houses

The successful control of silverfish that was achieved by the application of 10 per cent. DDT* in kitchens, carried out with the object of dealing with *Blatella* infestations, has already been mentioned. This led to further tests with this material.

No attempt was made in these tests to compare the efficacy of 10 and 5 per cent. dusts. It was appreciated that silverfish could probably be controlled by the use of a dust containing less than 10 per cent. DDT, but it now well established that a concentration of this order is required for the effective control of cockroaches, and therefore it is obviously desirable that dusts marketed for use against household pests generally should be standardized at the higher strength. Moreover, it is safe to assume that a 10 per cent. dust will remain effective for a longer period than a 5 per cent., thus permitting a longer interval to elapse between retreatments. As the labour involved in a thorough dust treatment far outweighs the cost of the material used, this consideration is of obvious importance.

The first test treatment was carried out in a heavily infested eight-roomed house. A total of 1½ lb. of 10 per cent. dust was blown into the crevices behind skirting boards, architraves, picture rails, &c., and into the spaces behind cupboards and stored lumber. Numbers of silverfish were driven out of their hiding places during the treatment, and several hours later many displayed characteristic symptoms of DDT poisoning. In the days immediately following the treatment, large numbers of dead silverfish were found on the floor of every room treated. A total of 483 were collected during the first two days; an additional 173 were counted in the third and fourth days, but after this the numbers declined, and two weeks after treatment there was an average daily count of only ten dead silverfish. The results of the treatment of this house are shown graphically in Fig. 1(A).

A series of tests was then arranged to determine the degree of control that could be expected if treatment was carried out by householders themselves, after receiving instructions on where and how to apply the dust. Altogether over forty houses were treated. It was hoped to obtain records of the numbers of dead silverfish picked up daily in each room over a period of four weeks after treatment. Most of the co-operating householders, however, ceased keeping records once it was evident that reasonable control had been achieved. The amount of dust used per house varied from 1 to 1½ lb., and the time of application was left to the occupants. From the results obtained it was evident that treatments carried out in daylight were as effective as those undertaken at night.

The records from those houses in which counts were maintained provide a good picture of the effect of the treatment. on infestations of varying degrees of intensity. The results obtained are represented in Fig. 1 (B—J). The numbers of dead silverfish counted, and recorded in Fig. 1, do not of course represent the total mortality caused by the dust treatment. Many insects must have died in crevices away from sight, and in several houses large numbers of dead silverfish were discovered behind lumber, &c., when this was moved some weeks after treatment.

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* Percentage calculated on p-p′ isomer basis.

In most of the houses treated, complete control of silverfish was attained, that is to say no evidence of any survivors was seen after a reasonable interval. In a few houses, however, small numbers of silverfish continued to be in evidence. It was noted that these were confined to the upper part of the building, i.e., the walls, &c., above the level of the picture rails, and the upper floor of two-storied homes. This observation led to an appreciation of the importance of ceiling treatment.
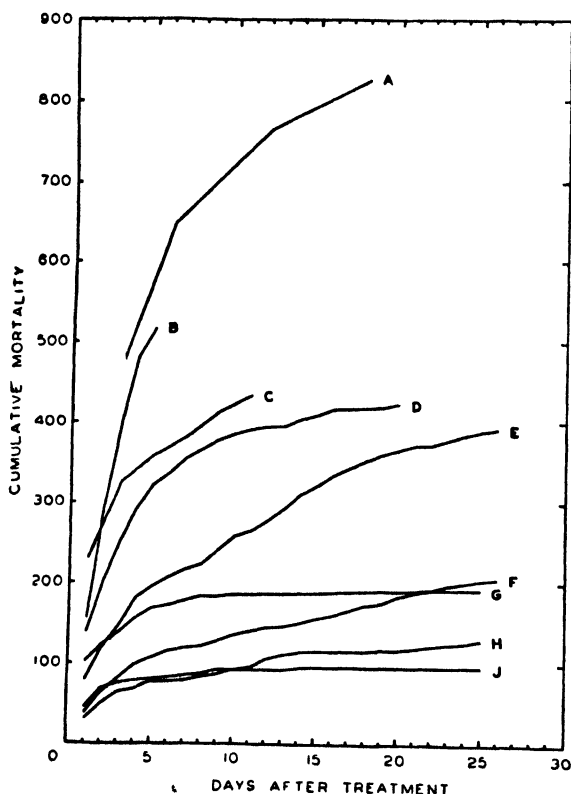
Fig. 1.—Cumulative mortality curves from silverfish-infested houses treated with 10 per cent. DDT dust.

After an interval of approximately three months, the houses in which silverfish were still in evidence were re-treated, with the same technique and the same amount of dust as were used in the original treatments. No marked mortality among the surviving silverfish resulted, and in two of the houses so treated silverfish continued to be seen in the upper parts of certain rooms. By this time it was suspected that the surviving infestation was harboured in the roof cavity. Accordingly, the upper surface of the ceiling of each house was treated, using ½ lb. of dust. This was followed by a sharp and substantial increase in the tally of dead silverfish, and then by the complete disappearance of the insects from the premises. The effect of one of these ceiling treatments is shown in Fig. 2.

4. Reinfestation from Wood Heaps

During earlier work connected with the use and distribution of bait cards, it was noted that heavy silverfish infestations in houses were usually associated with wood heaps, the collection of debris at the bottom of which almost invariably harbours a large silverfish population. A check in garages with wood heaps adjoining revealed concentrations of silverfish on the sides nearest the heaps. An analysis of mortality counts from treated houses showed that rooms with fires (kitchens, lounges, laundries) provided the highest tallies; in no house did a bedroom contain the maximum number of silverfish. Daily observations and counts of dead insects carried out in one house over a period of 55 days showed that healthy silverfish appeared after the replenishment of firewood in the kitchen, laundry, and bathroom (which contained a chip heater).
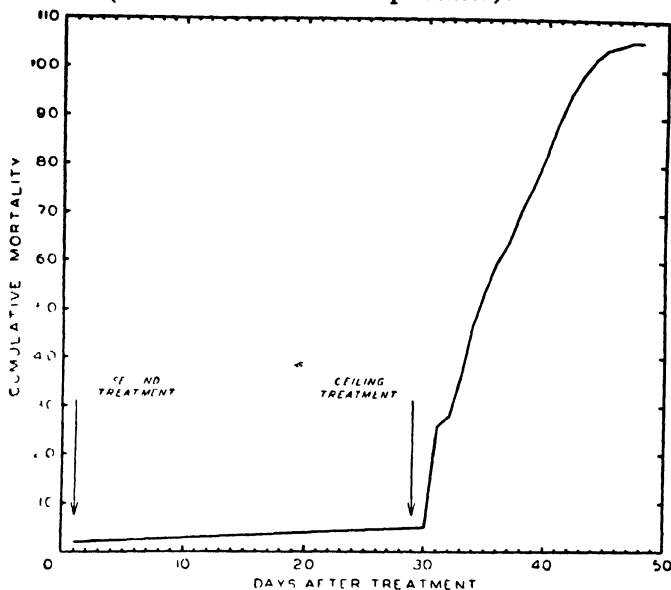
Fig. 2.—Cumulative mortality curve from a house in which the upper surface of the ceiling was treated with 1 lb. 10 per cent. DDT dust, after retreatment of the rooms had proved ineffective.

This evidence confirms that obtained by the author from an examination of many wood heaps over the past decade, and leaves little room for doubt that infested firewood provides a continual and important source of replenishment to the silverfish populations in Canberra houses. The author is satisfied that this problem has been completely solved in his own home by (i) clearing the wood heap site of all debris at least once a year, and (ii) blowing ¼-½ lb. of 10 per cent. DDT dust into the bottom half of the heap after restacking the wood.

5. Dust Treatment in Houses: Equipment, Technique

For the application of the DDT dust in a manner that ensures its effective penetration into the crevices where silverfish hide, some form of blower is essential. The small insecticidal powder applicators,

consisting of a rubber bulb and nozzle, can be used, but they are not really suitable. More satisfactory is a hand dust gun of the type shown in Fig. 3(A), which was used in the trial treatments described in Section 3. For the treatment of narrow crevices, such as those behind skirting boards and picture rails, with a minimum of mess and waste, a simple modification of the gun is desirable. This consists of a small metal fishtail tube, 2–3 inches long and with a slit opening ⅛-in. wide and about ⅝-in across, attached by a few inches of rubber tubing to the outlet of the duster (see Fig. 3). A makeshift fishtail can easily be made by cutting off the requisite length from the extension tube supplied with the duster, and compressing one end to form a slit. If the rubber connection mentioned above is replaced by flexible metal gas tubing the efficiency of the gun is further increased, as the connection is then sufficiently rigid to do away with the necessity of holding the fishtail nozzle with a finger when it is desired to direct the dust stream upwards or downwards.
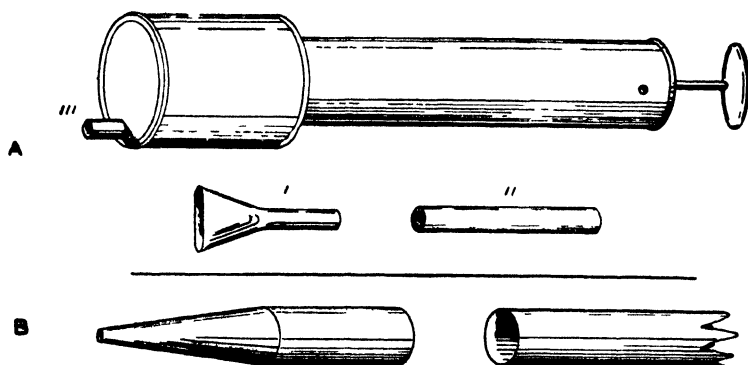
Fig. 3.—A.—Modification of hand dust gun. The metal fishtail outlet (i) is connected by a length of rubber tubing (ii) to the outlet pipe (iii) of the gun.

B.—Modification of discharge tube of knapsack duster showing attachment for reducing width of dust stream.

For large-scale operations a modified knapsack duster is preferable to the small hand gun. The discharge tube of this duster is shown in Fig. 3(B), and is described in the section dealing with the control of the German cockroach.

It must be emphasized that the test treatments described above, which resulted in such satisfactory control of silverfish infestations, were very thoroughly carried out. The object of the operators was to get dust into every crevice likely to harbour the insects. Each room was systematically gone through, and dust blown into the crevices behind skirting boards, architraves, and picture rails, not merely here and there, but at close intervals, and in such a manner that the dust was driven some distance along the crevice from each point of application. Special attention was also given to crevices

around fireplaces, and in built-in cupboards, &c.; and dust was blown into the spaces behind bookcases, wardrobes, and other pieces of massive furniture set close to the walls.

To treat the upper surface of ceilings, it is necessary, of course, to get into the roof cavity via the manhole. In a small house of simple shape it may be possible for an operator to distribute the dust sufficiently well while standing on steps with his head and shoulders through the manhole. Normally, however, it is necessary to go into the roof cavity and to blow the dust about so that it is well distributed on settling.

Examinations of, and reports from, treated houses indicate that a thorough treatment with 10 per cent. DDT dust, using the quantities and carried out in the manner indicated, should maintain a house virtually free from silverfish for a period of twelve months.

Most samples of 10 per cent. DDT dust handled by the Division of Economic Entomology have been practically odourless. Some, however, have had quite a strong odour, and their use in buildings has led to complaints by the occupants. It is desirable to make certain therefore, before treating a dwelling, that the dust to be used is free from any objectionable odour.

When treating rooms in which food is exposed or stored, precautions should be taken to prevent its contamination. Eatables should either be covered or temporarily removed.

6. Treatment of Offices

In some offices, and in certain parts of most of them, the use of DDT dust for silverfish control, such as is recommended for houses, is quite practicable. Officers of the Division have given demonstration treatments in one or two large Government buildings in Canberra, with, as far as can be ascertained, very satisfactory results.

In offices where the main infestation of silverfish, and the material that it is desired to protect from damage, is in filing racks, record cabinets, &c., that are in daily use, the application of dust is not desirable. For the control of silverfish in such offices, the use of a spray containing $0 \cdot 17$ per cent. pyrethrins and 4 per cent. DDT in deodorized kerosene was recommended to the Curator of Public Buildings, in the Department of the Interior, and is now in routine use.

The spray is applied with a power spray gun, fitted with a hook which allows the operator to hang the gun on his belt while climbing and moving the ladders required to treat filing racks. After blowing a fine spray into the spaces and crevices, to effect an immediate kill of the insects exposed or disturbed, the operator coats the tops and sides of the pigeon holes, &c., with the spray to leave a residual deposit of DDT. The efficacy of the residual treatment is indicated by reports of the appearance of dead silverfish weeks, and even months, after a building has been treated.

7. The Use of 10 per cent. DDT Dust to Control the German Cockroach

The efficacy of DDT against cockroaches is now well established and, as was mentioned in a previous section, the need for a dust containing a concentration of the order of 10 per cent. DDT for the control of the more resistant German cockroach is now recognized, chiefly as a result of accumulated practical experience in the United States of America.

At the time when the Division of Economic Entomology was called on to help deal with heavy infestations of the German cockroach in the kitchens of Service establishments erected in and near Canberra during the war years, information on the properties of DDT was still rather rudimentary. After a 5 per cent. dust had failed, in a preliminary trial, to give the degree of control desired, a 10 per cent. DDT dust was used in several large-scale treatments, with very satisfactory results.

For these treatments a knapsack duster was used, with the discharge tube modified to produce a fine stream of dust at increased pressure. The modification consisted of a conical metal attachment fitted over the end of the discharge tube, and tapered to a small hole of approximately $\frac{1}{4}$-in. diameter (see Fig. 3(B)). The junction between the attachment and the outlet tube was bound with adhesive tape to prevent the escape of dust. Modified in this way, the blower proved extremely effective: it was found that the dust stream could be driven a distance of 8–10 yards when required.

The technique of applying dusts for the control of cockroaches is essentially similar to that employed for silverfish control, i.e., it is blown into the crevices that provide the insects' daytime hiding places. Special attention, however, has to be paid to the crevices around ovens, hot-water containers, and pipes, where *Blatella* tends to congregate.

Between 3 and 5 lb. of dust was used in each of the kitchens treated. Many cockroaches emerged from their hiding places while the treatment was in progress, and within an hour they were distributed over the walls in considerable numbers. For several days after the treatment was applied very large numbers of dead and moribund insects were swept up from the floor. Immobilization apparently took some time, and the erratic movements of the insects affected by the DDT necessitated measures being taken to prevent them getting into food containers.

Discontinued occupation of many of the establishments prevented an assessment of the presistence of the effects of the treatments carried out. One of the kitchens treated with 10 per cent. DDT dust, however, has remained virtually free of cockroaches for a period of over two years.

A trial treatment with hexachlorocyclohexane (" 666 ") was carried out in one kitchen. A dust containing 5 per cent. of the crude material (0·63 per cent. of the gamma isomer) was used. Effective control was obtained, but the odour of the dust was so persistent and objectionable that the use of this material could not be recommended.

8. Acknowledgments

The author is indebted to those householders whose tallies of dead silverfish provided data for this paper. He is particularly grateful to Messrs. G. A. McIntyre and K. J. Prowse for their suggestions and enthusiastic collaboration in the treatment of their homes, to Mr. L. Lott for his co-operation in the treatment of Government offices, to Mr. D. G. Venables for assistance in assembling data and for the diagrams and graphs, and to Mr. F. N. Ratcliffe for assistance in preparing the manuscript for publication. Earlier work on silverfish control, during which some of the observations recorded in this paper were made, was carried out under Mr. G. F. Hill. Messrs. F. J. Gay and D. F. Waterhouse, who continued the laboratory studies initiated by Mr. Hill, were frequently consulted during the course of the experiments in practical control. Mr. Waterhouse was in charge of the team from the Division which undertook the treatments of the *Blatella*-infested kitchens.

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Recent Progress with Pelagic Fishing in Tasmanian Waters

By M. Blackburn, M.Sc., and A. M. Olsen, M.Sc.**

Summary

The Tasmanian pelagic fishing experiments, which were begun in 1941, reached a very promising stage in 1947. Two commercial-sized catches of horse mackerel were made by purse-seine under circumstances which suggest that much more can be achieved with larger gear. Lampara net catches of sprats were obtained more regularly and efficiently than before, and this work now has commercial prospects if conducted as an adjunct to other operations. Though limited, these results surpass any previously achieved in work of this kind in Australia.

Reference is made to many past difficulties which now seem to have been overcome. Such work requires the collaboration of skilled local fishermen, whose interest in it is now steadily growing.

Introduction

For several years it has been a major responsibility of the Council's Division of Fisheries to try to demonstrate commercial methods for the capture of Australian pelagic fish. In spite of the considerable abundance of suitable shoals in many areas at certain times, the work has been marked by very great difficulties. These arose out of certain unsuspected features of the fish occurrences themselves, inexperience with the methods, and latterly shortages of manpower and equipment. Some small successes were obtained at times, e.g. see Flett (1944), but these could not be repeated regularly. It was in 1947 that the experiments first achieved any degree of real success, and the following account is given for the possible guidance of others who will be doing similar work in the near future. It is hoped that the history of the Tasmanian project will indicate that progress can be made where there is perseverance, adaptability, and co-operation between all interested organizations. At present trade conditions are appropriate for pelagic fisheries development in various parts of Australia, especially south-eastern Australia, in a way in which they mostly were not before the war, and there are capable fishermen and merchants who are willing to co-operate in the necessary developmental work. Partly for this reason, pelagic fisheries development in Australia will now probably be much more rapid.

The developments in question were concerned with two species in south-eastern Tasmania, the horse-mackerel (*Trachurus novae-zelandiae*) and the sprat (*Clupea bassensis*). The former is known there as " mackerel " and this usage is followed here. Both species have been receiving attention from the Division for several years, and details of the preliminary catching experiments have been given in a previous report (Blackburn, 1943). As was shown in this report, these particular fish and their environment were by 1941 revealed to be more suited to offshore fishing with large surface nets than anything else encountered in the eastern Australian region, and a limited success was achieved in respect of purse-seining for mackerel in 1943, with two catches of which the larger was four tons. Although this

was very little it represented the best performance with any surface net in Australia to that date, and the only successful one by purse-seine.

The Mackerel Work, 1944 - 1946

For various reasons, no further advances were made, and even these limited successes could not be repeated in the three seasons following 1943. The optimum season for shoaling mackerel in these waters is from about February through to June. For the 1944 mackerel work, two boats operating different types of nets were used. The State fisheries vessel *Liawenee,* a 67-foot boat of the Danish-seiner class, worked during April, with a kind of purse-lampara net measuring about 330 by 12 fathoms. Mr. H. Watt's 56-foot ketch *Mary,* which had made the catches of the year before, used a purse-seine about 160 by 13 fathoms. This was some 40 fathoms longer than than one previously used, which was too short to encircle shoals regularly. Owing to the extreme shortage of netting in Australia, particularly in the finer mesh sizes, the net was made largely from 2½-inch mesh webbing with the addition of a limited quantity of finer material from the Division.

No success was achieved by either team, in spite of the presence of suitable shoals in adequate numbers. The men on the *Liawenee* found that the great length of their gear enabled them to encircle even fast-moving shoals, but the time required to purse a net with such a high length : depth ratio, without sinking the corks, was so long that the fish would get out. This difficulty had been foreseen, but it was thought it might be overcome by heaving in the wings very quickly by hand before pursing; however, in practice this was beyond a crew of nine men. There was some indication that the net might have worked well for fishing in smooth-bottomed waters within its own depth (as originally intended by its designers), but this did not altogether meet the circumstances of the mackerel problem and the work was not continued. The *Mary* worked much longer and set her gear oftener, but with the same result. The main trouble seemed to reside in the mesh size which allowed most of the fish to escape (one shoal got away completely in this manner after the net had been fully closed and pursed around it), but it also appeared that the net was still not long or deep enough to trap any but the quietest shoals.

After much discussion of these results it was decided that the ordinary purse-seine technique still promised favourably if better gear could be secured. The Council undertook to import a net, and specifications were prepared by those most closely associated with the work. An order was finally placed in the United States for a California-style purse-seine of 2-inch mesh and approximately 200 by 20 fathoms. From about this time onwards the Division of Fisheries initiated overseas orders for small lots of other nets that seemed likely to be useful for future pelagic fishing projects.

The net did not arrive until September 1945, and so nothing much could be done in the mackerel season of that year. Actually the *Mary* carried the old net for two months, but this was not expected to and did not lead to any very useful result. The season was more or less normal as regards fish occurrences.

For the beginning of the 1946 season the new purse-seine was carried by the *Mary*, which, as feared, proved not large or well-powered enough for such big gear, and after a short period it was transferred to the *Liawenee*. It was carried by her for a considerable time, but again there was intense disappointment, this time from a different cause. For the first time in at least six years, the shoals did not appear in their usual numbers, and there were virtually no opportunities for setting the net. One reason for concentrating so much effort on this particular resource had been that it seemed an unusually stable one, the order of abundance of the shoals being practically constant from year to year at the same season, which is not so with most of the other pelagic fish occurrences investigated in eastern Australia (this question has been discussed in detail by Blackburn and Tubb, unpublished). Naturally a non-fluctuating resource is to be preferred for a long-range series of fishing experiments. However, the 1946 season was definitely exceptional for these mackerel, probably not so much because of variation in their abundance in the area as because of a change in their availability at the surface where they might be sought by purse-seine, and it appears that this was mainly due to the very inclement weather at that time. Thus this season also passed without any success being achieved.

Unfortunately, the unsuccessful results caused a waning of interest in mackerel by the trade. Previously, Tasmanian fish canners and curers had been rather short of fish, but from about 1944 onwards they were able to obtain large supplies of barracouta, and mostly had more fish than they could comfortably handle. This turn in events was not really unexpected, since Tasmanian fishermen have always been more used to hook-fishing than netting in any form. Indeed one of the main troubles with the purse-seine work was the constant difficulty of obtaining men who could efficiently handle nets. It was hoped to obtain a skilled purse-seine operator from abroad, but in the outcome nothing was done.

The Mackerel Work in 1947

The situation was eventually relieved because of developments in New South Wales, where conditions were very different from those in Tasmania. Here, the immediate post-war period found fishermen and processors with not enough fish, instead of too many as in Tasmania. At the Narooma cannery there had been a shortage for years. On the fishermen's side, the abundance of " trawl " fish had not greatly improved from their war-time " resting " as was hoped, and indeed what was formerly the main species, tiger flathead, became scarcer than before. This affected the small-boat operators particularly, and some of them began to consider undertaking other kinds of fishing. Unlike the Tasmanians, many of them had had considerable net-fishing experience, including with large beach-seines for Australian salmon and other species, and it was natural for some to think about pelagic net fishing for the shoaling species of their own waters. The Division of Fisheries was in fact approached by two or three groups of fishermen for loans of gear for this work, which were all met as far as possible, but the details of only one such arrangement, the only successful one to date, are relevant here.

The vessel was the *Eden Star,* a boat of the Danish-seiner class (Fig. 1). She is 61 feet long with 18 feet beam and 9 feet draught, and has 102 h.p. with a speed of about 9 knots. The owner, Mr. W. Warn, borrowed the mackerel purse-seine from the Division in January, 1947, and spent 10 days in Tasmanian waters. An aerial reconnaissance located one extensive body of fish, but a change of weather prevented advantage being taken of this. Tests were then carried out near Eden (N.S.W.) for nearly two months.
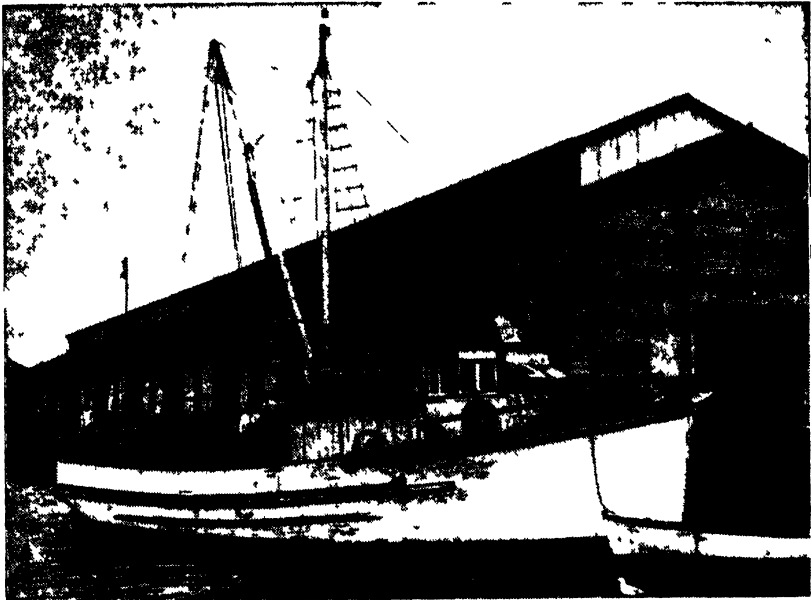
Fig. 1.—The *Eden Star* with purse-seine net   (Photo, Hobart "*Mercury.*")

The *Eden Star* and her crew encountered here all the main difficulties that the Division had met with in its own work on the mainland coasts over several years, namely, much unsuitable weather and wildness of the shoals offshore, inadequate depth in which to net the quieter inshore occurrences with this gear, and, at the last, the disappearance of most of the fish. When it was found that the purse-seine was too deep for shooting in Twofold Bay where many of the best occurrences were, the men built a purse-lampara or ring-net about 250 fathoms long, from material owned partly by the Division. With this they made one catch of 2 tons of bonito (*Sarda australis,* the "horse-mackerel" of New South Wales fishermen), which was in its way quite notable as being the first haul of any tuna species by surface seine in Australia. However, these fish took off before anything more could be done with them. Subsequently this net was dismantled to yield materials for lengthening the purse-seine by over 50 fathoms, after it was found that the latter would not encircle some of the fast-moving offshore shoals, but by the time these alterations were made the major fish concentrations had disappeared altogether. This made a return to Tasmania appropriate, and the vessel arrived there in the last days of March.

Mackerel were first observed a few days later in Monroe Bight. A very large shoal was encircled but it escaped by tearing away nearly all of the added lighter netting. Seals added to the damage, and considerable repairs were necessary. During the following 9 days' cruise the weather was somewhat unsuitable, and only one good occurrence of fish was encountered. This was in Fortescue Bay, in the vicinity of the previous attempt, and there was again interference by seals, which caused the selected shoal to submerge as the net was about to be set. Darkness fell within a few minutes (these mackerel often shoal at their best in the last hour of daylight) and there was no chance to manoeuvre for a second attempt. The fish were not found here thereafter, or elsewhere, and an aerial survey of the whole south-eastern Tasmanian area also failed to locate any significant occurrences at this time. It might be observed that those responsible for the earlier tests had had little regard for the east coasts of the Forestier and Tasman Peninsulas (including the localities mentioned above) for this work, in spite of frequent mackerel occurrences at times. This was because of seals which are particularly numerous there, and also the very strong currents which tend to confine any surface netting activity to the one or two available bays.

The next cruise was in the Maria Island area, and on April 23 a suitable shoal was located and followed for almost 2 hours before a favourable opportunity for shooting the net occurred, at 4 p.m. This shot was very successful and yielded about 15 tons of mackerel averaging 2½ lb. The locality was off Oakhampton Bay in about 22 fathoms, and the sea was calm and general conditions ideal. Six days later the second catch, of about 16 tons, was made near Cape Bernier, at about the same hour and under similar weather conditions. In the early afternoon several shoals were found close inshore, but the vessel managed to work one of the larger ones into the deeper water where the purse-seine could be used. During the pursing some more of the lighter netting, which had again been added to increase the length of the net, tore under the weight of fish. In consequence a large part of the catch was lost, leaving only the 16 tons which were actually got aboard. These fish averaged about 2 lb.

The fourth cruise of the series commenced on May 5, after repairs to the net had been effected. The *Eden Star* returned to the Maria Island area and had no trouble in locating fish, which were also found here in the course of an aerial reconnaissance during the same period. Five sets of the net were made but were all unsuccessful. What had been suspected on some earlier occasions was now established, that the gear as imported was too short to encircle the most active shoals, at least without frightening them so much that they would submerge before the net was pursed. It was found in earlier years that the shoals often become livelier, and do not stop up so well at the surface, towards the end of the season. However, the successes of the *Eden Star's* two April hauls were probably also due to the lengthening of the net, whereas there was no material left for this on the fourth cruise. Finally the net was measured and found to be only 170 fathoms long instead of the 200 specified. The Division then made available some old netting it had in Hobart, and the length was again increased to about 200 fathoms. A fifth cruise was made for a few

days at the end of May near Maria Island, but the shoals found were quick and erratic in their appearances and the two shots made were unsuccessful. The *Eden Star* carried six men throughout her work.

Comment on the Mackerel Work

The two purse-seine catches made by the *Eden Star* represented the first truly commercial-scale hauls of any pelagic fish by any surface net anywhere in Australian waters. Had this performance been repeated even once or twice more the whole question of the suitability of the method in this particular case would have been at an end, but what has actually been achieved is in itself extremely promising. One might be more doubtful if there was no reasonable explanation for the seven subsequent failures, or if it was not practically possible to correct what was at fault on these occasions, but this is not so. It is quite plain that a net 170 fathoms long is too short to take the fish regularly (which incidentally indicates that the three-man crew of the *Mary* did remarkably well to take the fish they did in 1943 with 120 fathoms), but that a longer one is suitable for this purpose. As shown above, it was always thought after 1944 that 200 fathoms would be necessary, but the crew of the *Eden Star* now believe that as much as 250 fathoms is required to cope with all eventualities. As was emphasized in the published 1943 report, one has to consider the depth of water also in connection with the lengthening of the net, because if the length : depth ratio very greatly exceeds 10:1 the corkline will sink and release the fish in the pursing process, unless this is so slow as to run the risk of loss of fish by their submergence. However, although mackerel may shoal in 10 fathoms of water or less, the experience of many seasons' work now indicates that schools also occur fairly often in waters of between 20 and 25 fathoms, though much less commonly further out. Therefore (bearing in mind also that purse-seining can be carried out in shallow water if the bottom is smooth) a net of maximum length 250 fathoms and minimum depth 20 fathoms would probably meet all the requirements of this work. The maximum ratio in this case, 12½ : 1, is high, but it is found in some commercial Californian nets (see Fry, 1931, p. 19). At the time of writing the Division of Fisheries is assisting Mr. Warn to enlarge the present gear on these lines, for further work in New South Wales and Tasmania.

The question of what actually constitutes economic fishing depends on operating costs and available prices for the fish, which may both vary considerably. However, it can be said, for example, that an average catch of 15 tons per fortnight, i.e., much less than obtained by the *Eden Star* in its period of success, would at present prices constitute a very good return for a boat and crew such as was operating in this case.. Even if prices fell, as they might if such quantities were regularly landed, there would still be some margin. However, there is reason to suppose that, with proper gear as suggested above, the rate of catch would be higher than this. The experience of the *Eden Star's* second haul suggests that quantities well over 15 tons could probably be taken in a single catch at times, and, furthermore, it is certain that opportunities for setting the net, on large shoals under good conditions, would occur oftener than

fortnightly. In the unpublished report by Blackburn and Tubb mentioned above, the records of Tasmanian mackerel shoals of several seasons were very fully analysed, and it was shown that sizeable concentrations suitable for purse-seining were liable to be found on an average of 1 in every 5½ days, over the 5-month season February to June, in nearly every year. Allowing for some shots being failures, and for occasional bad seasons such as 1946, it appears nevertheless that the work might prove very profitable. At any rate it has been shown that the method is quite feasible and will yield large catches, and, although a demonstration of continuous catching has yet to be made, there is no longer much doubt, in the mind of anyone associated with the work, that it will eventually prove a sustained success in Tasmanian waters. Tests under proper conditions will shortly be carried out in New South Wales waters. In both States there is now a considerable demand for good quality canning fish.

Fig. 2.—Stern of the *Eden Star* with purse-seine ready for use. Note rings over port side, with purse-line running through from large coil on deck (Photo, A M. Olsen.)

Fig. 2 shows the stern of the *Eden Star* with the purse-seine ready for use. It will be seen that there is no turntable and that the winch is of the ordinary " Danish-seine " type. An interesting point is that the purse-rings hang together over the side and the purse-line runs through them from a large coil on the deck. This is not, or at any rate was not formerly, the Californian practice with purse-seining, although something very similar is done in respect of ring-net work (see Fry, 1931, pp. 40–2). Thus the purse-line and purse-rings of such nets that were carried on the Division's vessel *Warreen*, from 1938 to 1942, were always piled in with the leadline following Californian practice, and this gave constant trouble with the kinking of the purse-line, so that there were many times when pursing could not be completed. When Mr. T. Challenger and the senior writer commenced their operations in 1942 the other method was tried and proved an immediate success, so that it has been used by all crews since in this Tasmanian work.

The localities on the south-east Tasmanian coast that have been found particularly good for mackerel shoals, leaving aside some that do not lend themselves very well to purse-seining as noted above, are from north to south Cape Lodi, Wineglass Bay, Schouten Passage, Oakhampton Bay, Darlington, Reidle or Halfmoon Bay, Cape Bernier ("Hellfire Bluff"), Green Island, Fortescue Bay, Wedge Island, and Adventure Bay. The more landlocked waters, such as the D'Entrecasteaux Channel, and Frederick Henry, Norfolk and Blackman's Bays, are poor areas. Mackerel also occur north of Cape Lodi but seem less conspicuous there in shoals.

The Sprat Work

Concurrently with the mackerel effort, the Division and its collaborators have continued to devote some attention to the question of fishing Tasmanian sprats. As a result of the 1947 trials in particular, this project can now be regarded as satisfactorily established from the trade side, although no very high level of output can reasonably be looked for here. The fish are true sprats which are very closely related to those of that name that support a sizeable fishery in the North Sea and the Baltic, and which before the war were available in Australia as canned "sardines" or "sild" from Norway (see Blackburn, 1941, p. 52). Apart from a probably erroneous record from South Australia the species has not been reported outside Tasmanian waters. It has been found as far north and west as Flinders Island and the Tamar River, but in the experience of the Division's officers it is most abundant in Storm Bay and adjacent waters. Actually, although shoals have been reliably reported elsewhere, the only ones seen in the course of the organized survey work have been in the southern waters of the D'Entrecasteaux Channel. The limits of the season there are not precisely known, but probably the surface shoals are most numerous from about March to June. At this season, it has been found that good concentrations are liable to be found on about one day in nine on an average, although there is some slight annual fluctuation (Blackburn and Tubb, unpublished).

These sprat shoals were first noticed by the Division's workers in 1940, when the *Warreen* attempted to catch them by lampara net. These attempts failed, but in the following year there were a few significant catches, including one of about 2½ tons. A portion of this particular haul on the Hobart wharfs is shown in Fig. 3. The main interest in the species up to then had been in their possible utility for live bait for tuna, but it was found that the fish could not be held alive for this purpose after netting, because of excessive loss of scales (see Flett, 1944). However, it was then considered that they might serve for canning, and when the special co-operative efforts with the Tasmanian Fisheries Division began in 1942 these fish received more attention than mackerel for a time. Further lampara work in that year resulted in a few more hauls of significant size, and it was decided to attempt to catch them by purse-seine in 1943. As shown elsewhere (Blackburn, 1943) these trials did not succeed, partly because of faults in the net but partly also because of relative scarcity of suitable shoals. The main effort was immediately turned to mackerel and proved more successful with that

fish, with the result that the mackerel work has since always been regarded as potentially much more important. The 1943 work also emphasized the localized distribution of the sprat and the very moderate extent to which it occurred in sizeable surface shoals, and it was not long afterwards that it ceased to be regarded as likely to give rise to a substantial independent fishery, by any of those associated with the tests.

Fig 3—Portion of catch of sprats made by the
Warreen (Photo, J A Tubb)

However, the matter was not lost sight of in the following years. It had at least been established that the fish could be caught with some regularity by lampara net, even though the catches were not large and the opportunities for making them not very frequent. Some of those who had participated in the experiments realized that sprat fishing might at least be profitable if conducted as an adjunct to other fishing operations (such as for barracouta and scallops, which are taken in the south Channel waters at the same season), especially if the fish could be sold for curing, thus commanding a higher price than would be economic for canneries. Practically nothing was done in 1944, but in 1945 and 1946 private operators conducted some more tests with the lampara and other gear provided

by the Division. The catches obtained were small, averaging only a few hundredweight each, but there were nearly always some fish taken. The lampara crews of four men were smaller than at any time before, but this number was still uneconomically large for what was visualized as an essentially part-time occupation for small boats.

In the end it was the general opinion that the lampara was the appropriate gear, since it was small and light and could be stowed away when not required, and brought out, used, and put back without much trouble when sprat shoals were encountered. What seemed to be required was a special lampara with relatively short wings, which would not need so much manpower to haul aboard before the fish could escape. Fortunately the Division had previously placed an order for some such nets in California, and when these arrived in 1946 one was allocated to Mr. T. Sward for use in the following sprat season on his vessel *Valetta*. These nets were about 150 fathoms long, whereas those used previously had been about 200 fathoms. The sprats had for years been remarked as quieter in the shoaling state than any other closely investigated Australian pelagic species, so that the question of length of net to encircle them was not so important.

This net was carried for about seven weeks in March and April, 1947. The area of operations was again the south part of the Channel. It is not clear how many days were actually devoted to searching for shoals, but there were only three on which they were found in good quantities. However, on each of these there were three hauls of the net around sprats, the total daily captures being respectively 2,069, 2,447, and 1,833 lb. There were two negative sets and the other individual catches ranged from about 300 to 1,833 lb. These were of much the same order as obtained previously, but the important points are, firstly, the regularity with which fish were obtained, and, secondly, the small number of men in the crew. This was three, except for one haul for which only two were available, but which, nevertheless, yielded about 750 lb. The operators consider that these results indicate real, if limited, possibilities with sprats for commerce. The writers are informed, by representatives of a leading food processing firm which is using these fish for paste manufacture, that they are particularly suitable for that purpose because of their very fine flavour. This is said to surpass that of the real anchovies and pilchards of Australian origin that are being used for this process by the same company. There are also other possibilities on the marketing side, and with a satisfactory fishing technique available the question of developing the sprat resource can be left to the enterprise of the fishermen and processors.

Acknowledgments

The successful results obtained in 1947 were only made possible by the collaboration of the Tasmanian Fisheries Division, and of various private operators, with the Council's Division over a number of years. Mention has already been made of the work of Messrs. W. Warn, H. Watt, and T. Sward, and it is also desired to thank all the members of the crews of their vessels. The work on the *Liawenee* was carried out by Captain H. Johnston, Mr. A. Burnett, and also

Mr. W. Warn, who was mate for a period in 1944. Dr. H. Thompson was in the last instance responsible for what was done by the Council's Division of Fisheries, and he was especially concerned with the organizing of the successful 1947 work. Mr. S. Fowler was of considerable assistance on the aerial reconnaissance side in various years, the junior author also participating in this in 1947. Mr. J. A. Tubb gave much useful advice and encouragement at all times, and Mr. W. S. Fairbridge supervised some of the boat work that was done from 1944 to 1946. Probably no one did more throughout than Mr. T. J. Challenger, Tasmanian Inspector of Fisheries, but for whose practical fisheries capacity the work would certainly not have started under the difficult conditions that existed during the war, and whose enthusiasm and patience were very largely responsible for keeping going at times when the prospects seemed almost hopeless.

In conclusion, it is desired to thank the proprietors of the *Mercury* newspaper of Hobart for permission to reproduce the photograph of the *Eden Star*.

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Addendum

Since the above was written, the *Eden Star* has made two catches of 18–20 tons of the same "mackerel" in New South Wales waters (October and November 1947). This followed the lengthening of the purse-seine to 235 fathoms.

Laboratory Methods for Testing the Resistance of Textiles to Attack by Fungi

*By G. C. Wade, M.Agr.Sc.**

Summary

The pure culture tests used for testing mould resistance of textiles are discussed. *Memnoniella echinata* is used for testing resistance to loss of tensile strength, and *Aspergillus niger* for testing resistance to loss of waterproofness due to micro-organisms.

Test samples are sterilized by exposure to methyl alcohol, which is removed by evacuation in a desiccator or vacuum oven. Samples are then rested on mineral salts and agar medium, inoculated, and incubated for 14 days at 30°C.

Soil burial tests are also described. Greatest loss of strength occurred at 80 per cent. of the water-holding capacity, but for routine tests a moisture content of 65–70 per cent. was used. The relative merits of the two types of methods are discussed.

1. Introduction

With the outbreak of war in the Pacific, it became obvious that Australian troops would be required to fight in tropical areas, and that materials of an organic nature, including cellulosic fabrics, would be liable to deterioration through micro-organisms under those conditions.

It was therefore necessary to develop protective treatments, and rapid laboratory methods of testing such treatments were investigated as part of this programme. Two main classes of test methods, namely, pure culture tests and soil burial tests, had been used previously in overseas work and both these methods were studied here, in collaboration with the General Chemistry Section of the Munitions Supply Laboratories, Maribyrnong, Victoria.

2. Pure Culture Tests

The principle of these methods is to inoculate the fabric under test with a pure culture of an organism known to cause deterioration of untreated fabric, under conditions favourable to the organism, and after inoculation to determine the effect of the organism on the treated fabric or, in some cases, note the amount of the growth of the organism.

Thom *et al.* (1934) developed a method in which the fabric, after sterilization by autoclaving, was rested on a sterile medium of mineral salts and agar and inoculated by a pipette with a spore suspension of *Chaetomium globosum.* After incubation at 28°–30° C. for 14 days, the tensile strength of the fabric was determined. Subsequent investigators (Furry *et al.*, 1941; Greathouse *et al.*, 1942; Rogers *et al.*, 1940) have changed details of the method, but it has formed the basis of subsequent pure culture tests.

Following work first conducted by Dr. H. L. Jensen in collaboration with the Munition Supply Laboratories, it was found that a number of surface moulds are also of importance on tentage since they reduce waterproofness of the fabric, and test methods have been developed in the course of this work to test efficiency of treatments against these fungi, as well as against cellulose-digesting forms.

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* Now an officer of the Tasmanian Department of Agriculture; the work described was carried out while the author was on the staff of the Victorian Department of Agriculture and a member of the Mycological Panel of the Scientific Liaison Bureau.

(1) *Testing Resistance to Attack by Fungi Which Reduce Tensile Strength*

The method of Thom *et al.* (1934) was used in the early work, except that *Stachybotrys atra* was used as the test organism and the inoculation was conducted with an atomizer instead of a pipette. *Stachybotrys atra* has also been used by New Zealand workers (Brien and Dingley, 1946). Cultures of the strain of *Chaetomium globosum* used in America were not available in Australia at the time, and *S. atra* was a more powerful cellulose digestor than any of the *Chaetomium* species then tested. *Memnoniella echinata* subsequently replaced *S. atra* in our tests since it was more common on deteriorated cellulosic materials returned from the service areas, and had similar activity as a cellulose-digesting organism. Greathouse *et al.* (1942) used *Metarrhizium glutinosum* in their work.
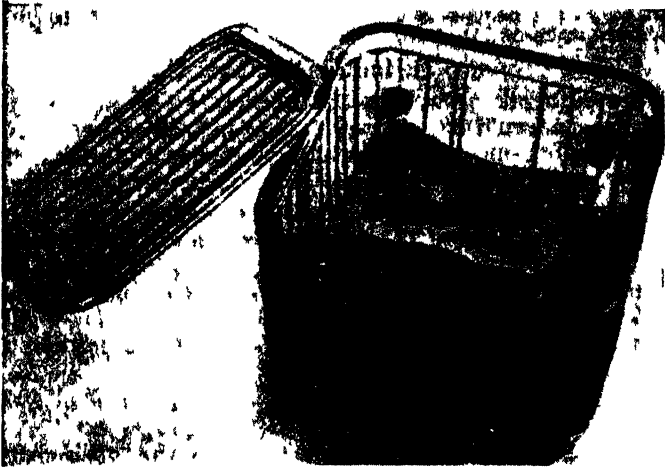
FIG 1—Showing refrigerator box containing mineral salts and agar medium, and strip of untreated duck which is held in place by single cotton thread

FIG 2—As Fig 1 after cutting cotton thread and pressing duck down on to agar.

Inoculation with a mixture of fungi was not used as it was demonstrated that many of the fungi concerned were antagonistic to one another. In one experiment to check this, untreated cotton duck was inoculated with *Memnoniella echinata, Stachybotrys atra, Chaetomium* sp., and with combinations of the fungi. It was found that if *M. echinata* was present in a mixed spore suspension it became dominant and other fungi were excluded. If duck was inoculated with a mixture of *Chaetomium* and *Stachybotrys* both fungi grew in well-defined areas and did not invade areas already attacked by the other organism. Tensile strength determinations showed that strength of duck inoculated with a mixture of *M. echinata* and *Chaetomium,* or with a mixture of *M. echinata, S. atra,* and *Chaetomium,* was significantly greater, after incubation, than of duck inoculated with *M. echinata* alone.

In order to determine whether the antagonism was due to toxic materials produced by the fungi, an experiment was conducted in which *Aspergillus niger, Chaetomium* sp., *Penicillium luteum, M. echinata* and *S. atra* were grown on Czapek's solution for 14 days. The cultures were then filtered through a Seitz filter and added to Czapek's solution, a similar volume of distilled water being added to another series as a control. Flasks containing each filtrate were inoculated with *A. niger, P. luteum* and *M. echinata.* After eight days' inoculation, the fungal mats were filtered off, dried, and weighed. It was found that *Stachybotrys atra* produces a toxic principle which inhibits the growth of *Aspergillus niger* and *Memnoniella echinata,* and *P. luteum,* a principle which inhibits growth of *A. niger.* Thom *et al.* (1934) and Barker *et al.* (1943) have also found the use of mixed inoculations unsatisfactory.

Refrigerator boxes of the type shown in Figs. 1 and 2 were used. After preparation, the mineral salts medium was distributed to the boxes to give a depth of about ¼ in. in each and two glass rods with pieces of rubber tubing on each end, to prevent slipping of the rods, were placed across the box to hold the ends of strips off the agar (Fig. 2). In preliminary work on the influence of the medium, etc., two strips of untreated fabric each 11 in. by 2½ in. were folded over the rods and held in place by a single stitch of cotton (Fig. 1) and the whole sterilized by autoclaving for 30 minutes at 18 lb. pressure. After autoclaving the thread was cut and the strips pressed down in contact with the agar (Fig. 2). In tests of treated fabric, the boxes containing media were autoclaved and the fabric separately sterilized by methyl alcohol, as described later.

The work was conducted using Thom's (Thom *et al.;* 1934) medium of the following formula (Medium A):—

Sodium nitrate	3·0 g.
Potassium phosphate (K₂HPO₄) ..	1·0 g.
Potassium chloride	0·25 g.
Magnesium sulphate	0·25 g.
Agar	10 g.
Water	1,000 ml.

This medium was compared with that of Greathouse *et al.* (1942) (Medium B).

Potassium phosphate (K_2HPO_4)	..	1·394 g.
Magnesium phosphate	.. ..	0·740 g.
Ammonium nitrate ..	.. ..	1·001 g.
Calcium carbonate ..	.. ..	0·005 g.
Sodium chloride ..	.. ..	0·005 g.
Iron, zinc, manganese as sulphates	..	0·001 g.
Agar	..	10 g.
Water	..	1,000 ml.

It was also compared with a medium of distilled water and agar. Mercerized cotton of a breaking strain of 80 lb. per inch width was used for the tests, which were conducted as described above. *Stachybotrys atra* was used as test organism and the tests were incubated for seven days at 30°C. All the tensile strength determinations described in this work were made at the Munition Supply Laboratories, Maribyrnong, using a Goodbrand tester. Six replicates were used. The results are shown in Table 1.

TABLE 1.—EFFECT OF COMPOSITION OF THE MEDIUM ON THE DETERIORATION CAUSED BY S. *atra*

Medium	Mean Tensile Strength Per Inch Width	pH of Medium*
Agar-distilled water	29	6·0
Medium A	3	7·5
Medium B	5	6·4

* Original pH 7·0.

The analysis of variance as described by Snedecor (1940) was applied to the results.

Difference for significance at 5 per cent. level = 3 lb.
Difference for significance at 1 per cent. level = 5 lb.

This test demonstrated the accelerating effect of a mineral salts medium. There was no significant difference between the results with the two mineral salts media and, since much information had already been obtained with medium A, its use was continued.

To determine the influence of the quality of agar on the test, medium A was prepared using two different samples of powdered Japanese agar, a sample of shredded Japanese agar, and agar of Australian manufacture. The test was then conducted in the usual way, using twelve replicates of mercerized cotton of initial breaking strain of 80 lb. per inch width. They were inoculated with S. *atra* and incubated for seven days at 30°C. The results are shown in Table 2. All the samples of Japanese agar proved equally effective, but the Australian

agar was unsatisfactory, because it gave an inferior gel and the variability of the results as shown by the standard deviation was considerably greater than with the other agars.

TABLE 2

Agar Used			Tensile Strength	Standard Deviations
Powdered Japanese agar A	..	..	12·7	3·2
Powdered Japanese agar B	.	..	9·6	2·2
Shredded Japanese agar	..	..	13·6	2·6
Australian agar	..	..	19·5	13·5

Difference for significance at 1 per cent. probability level = 7·2 lb.
Difference for significance at 5 per cent. probability level = 5·4 lb.

Agar was in short supply during the war and Greathouse *et al.* (1942) suggested using a solution of mineral salts with a wick of glass fabric to support the test material. Glass fabric was not available in Australia, but tests were conducted using glass tape wound on a broad U-shaped glass rod, and with washed sand, watered with Thom's mineral salts solution. The strips lost less strength on sand than on agar. The loss of strength on the glass tape frame was not significantly different to the loss on agar, but the results were more uniform. However, the method was not adopted as it was found that the frames could not be cleaned adequately for re-use, and the labour of preparing them was prohibitive for routine tests.

To determine the relative efficiency of inoculation with a pipette and an atomizer, mercerized cotton was laid on medium A in refrigerator boxes in the usual way, and twelve strips inoculated by a pipette and another twelve by a de Vilbis atomizer, using a spore suspension of *Stachybotrys atra* containing 670,000 spores per cc. After incubation for seven days at 30°C., the tensile strengths were determined, and it was found there was no significant difference between the results obtain by the two methods.
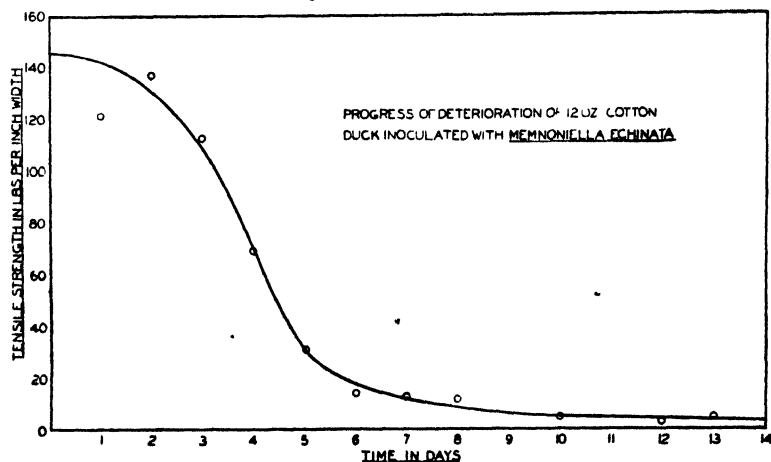
FIG. 3.—Relation between time of incubation on extent of deterioration of 12-oz. cotton duck inoculated with *Memnoniella echinata.*

To determine the effect of concentration of the spore suspension, mercerized cotton strips were inoculated with suspensions of S. atra containing 670,000, 380,000, and 120,000 spores per cc. It was found that within these limits the concentration of the suspension had no influence on the results of the test.

To determine the effect of time of incubation on loss of strength of cotton duck inoculated with *M. echinata* and *S. atra,* untreated duck was rested on medium A and four strips removed after each day of incubation and the tensile strengths obtained. The results for *M. echinata* are shown graphically in Fig. 3. With both organisms the loss of strength had reached a maximum in fourteen days and this was adopted as the time of incubation for routine tests.

Tests with treated materials were carried out on the material as received, and after washing in a water spray for seven days (three and a half days each side). It was considered desirable that the leaching should be as severe as possible; the seven-day period was the maximum that was practicable for a routine test. The variation in temperature of the water spray was unfortunately high (17° $\pm$ 5°C.), but there is no evidence that this variation is of great significance.

The volume of work necessitated a restriction in the number of test pieces, and experimental work suggested that sufficient data for practical purposes could be obtained from the inoculation of three strips in each test.

It was not always possible to determine the tensile strength of strips immediately after incubation, and they were therefore sterilized by immersion in methylated spirits.

(ii) *Resistance to Attack by Fungi that Reduce Waterproofness*

Work by Dr. H. L. Jensen (1946) in collaboration with the Munitions Supply Laboratories showed that certain Aspergilli and Penicillia were able to attack copper soap-treated fabrics and cause loss of waterproofness, and reports from New Guinea indicated that loss of waterproofness of untreated tentage due to attack by similar fungi was a serious problem. Therefore, in addition to testing for resistance to cellulose-digesting organisms, it became standard practice to test also for resistance to waterproofness-destroying fungi.

Other workers, e.g. Marsh *et al.* (1944), have tested the resistance of treated fabrics to species of *Penicillium* and *Aspergillus,* but have relied on noting the amount of growth, and do not refer to the effect of the organisms on waterproofness. The work conducted here has shown that the determination of the effect of attack of the organism on the fabric gives much more reliable results than noting the surface growth.

Aspergillus niger was used as test organism since it was commonly found on both treated and untreated fabrics and caused serious loss of waterproofness. Four circles, 4 inches in diameter, were sterilized with methyl alcohol vapour, and then pressed down on to medium A in petri dishes. After inoculation they were incubated for fourteen days at 30°C., and the waterproofness determined at Munition Supply Laboratories, Maribyrnong, using apparatus developed there, which measured the head of water at which the fabric first showed leakage. In some of the early work, the circles were sterilized with methylated

spirits after incubation, but it was found that this practice led to unreliable results, as impurities in the liquid reduced the waterproofness of the fabric. Also, elimination of the use of methylated spirits obviated any possibility of redistribution of water-repellent substances. In practice, therefore, the test samples were dried out after incubation, and the waterproofness determined as soon as possible.

The amount of macroscopically visible mould growth was also noted, but it was found that there was little correlation between it and the loss of waterproofness.

(iii) *Sterilization Technique*

The use of a sterilization treatment in the testing procedure is necessary to avoid the growth of organisms differing in activity from the test organism and possibly antagonistic towards it.

In overseas work, sterilization by autoclaving has been adopted generally. It is obvious that autoclaving is unsatisfactory when dealing with fungicides that have a high solubility in water or are volatile, and it became evident that autoclaving may also alter the distribution of many non-volatile fungicides that have a relatively low melting point, e.g. copper soaps. Autoclaving can thus only be safely used with a few materials, such as those treated by the cuprammonium process.

It was therefore necessary to examine other methods of sterilization. Schweizer (1931) developed a method of sterilizing culture media, in the cold, by use of volatile fungicides. He listed various volatile fungicides that were effective and described suitable apparatus for sterilization. Very much earlier Krais (1919) used chloroform vapour to sterilize nettle stems in retting experiments. Experiments were therefore conducted to develop a method of using volatile fungicides for sterilization in textile testing. Methyl alcohol was included in the tests as Dr. S. D. Rubbo, Professor of Bacteriology, University of Melbourne, was using the vapour of this material for sterilization of optical instruments.

In the first experiment, 2-in. square pieces of 18-oz. cotton duck were inoculated with a spore suspension of *S. atra*. Four pieces of inoculated duck were immediately placed on the mineral salts and agar medium previously described. Four pieces were placed in a sealed jar containing a layer of methyl alcohol, four in a jar containing xylol, and four in a jar containing chloroform. After exposure to the vapour for three hours at room temperature, the pieces of duck were removed with sterile instruments and placed on mineral salts and agar medium. All samples were then incubated at 30°C. for seven days, when they were examined for growth of the organism. Vigorous growth developed from the unexposed pieces and the pieces exposed to xylol. Methyl alcohol and chloroform gave complete sterilization.

Chloroform was unsatisfactory because of its softening effect on some finishes. The vapours of both chloroform and methyl alcohol appeared to be absorbed by the fabric. Because of its lower solvent properties, methyl alcohol was preferred and it was considered that the amount absorbed was insufficient to affect substantially the distribution of any fungicide or water-repellent substance. Further experiments were conducted at various temperatures to determine the

minimum time required for sterilization by methyl alcohol of duck in which *S. atra* had commenced growth. In this series of tests, 18-oz. untreated cotton duck was inoculated with *Stachybotrys atra* and incubated for 48 hours at 30°C. to enable the fungus to germinate and penetrate the duck. The duck was then exposed above methyl alcohol at 20°, 30°, 40°, and 50°C., and pieces removed at hourly intervals and placed on mineral salts and agar medium and incubated. Under these conditions, sterilization took over 6 hours at 20°, 6 hours at 30°, and was effective in 1 hour at 40° and 50°C.

It seemed possible that sterilization would be less effective with fabric treated with chemicals, such as copper soaps, which would hinder penetration of the volatile fungicides. A test was therefore carried out in which 12-oz. cotton duck, treated with copper soap, was inoculated with *Aspergillus niger*. It was then incubated in a humid atmosphere for 48 hours. Pieces of the duck were then exposed to methyl alcohol and chloroform vapour respectively at room temperatures and at 40°C. and the minimum time for sterilization determined. Methyl alcohol was effective in three hours at room temperature and in one hour at 40°C. Chloroform was effective in three hours at both temperatures.
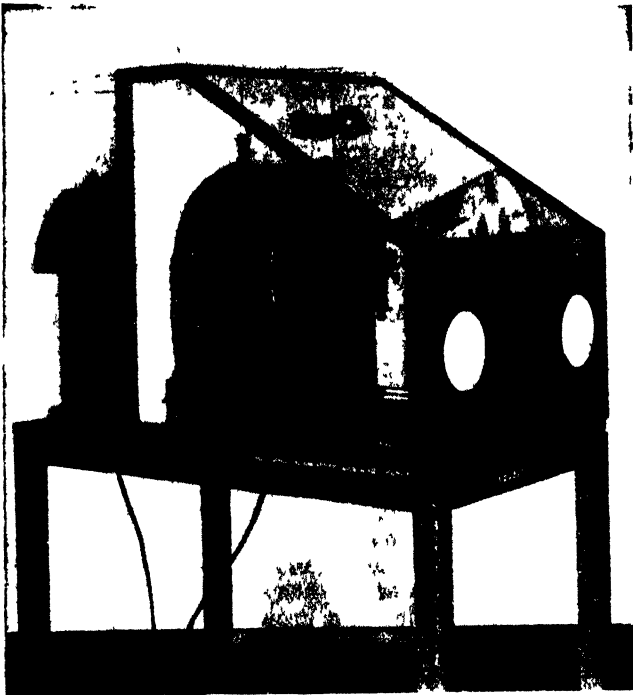
Fig. 4.—Transfer chamber with vacuum oven in position.

Strips of untreated duck retained sufficient methyl alcohol after exposure to inhibit growth of *Stachybotrys atra*. It was found that the methyl alcohol could be completely removed by transferring the fabric, after exposure, to a sterile desiccator, which was placed in an oven at 40°C. and evacuated for one hour.

The time of exposure to methyl alcohol adopted for routine tests was a minimum of four hours at 40°C. or a minimum of twelve hours at room temperature. The transfer of fabrics from the vessel containing methyl alcohol to the sterile desiccator, and the transfer to the refrigerator boxes containing the mineral salts and agar medium was carried out in a transfer chamber of the type shown in Fig. 4. The transfer chamber was first sterilized by atomizing propylene glycol on to an electric hot plate, maintained at a temperature of approximately 100°C. (Wade, 1947).

A simplified technique, which eliminates the transfer of strips from one desiccator to another, was subsequently developed. The strips to be sterilized are arranged loosely in a horizontal vacuum oven, fitted with a metal barrier so placed that the floor of the oven acts as a tray, and an outlet pipe flush with the floor of the oven. Methyl alcohol is poured into the bottom of the oven, which is then closed. The pressure is slightly reduced, and the temperature raised to 40°C. After four hours the pressure is restored to atmospheric by passing in air, which has been filtered through cotton wool. The outlet valve is opened and the methyl alcohol run out. The valve is then closed, the oven evacuated and maintained at 40°C. for one hour. After restoring the pressure to atmospheric, the oven is opened and the strips transferred to autoclaved refrigerator boxes containing the mineral salts and agar medium. This latter operation is carried out in a transfer hood which fits on to the end of the vacuum oven.

3. Soil Burial Method

According to this method, the fabric under test is buried in soil of definite moisture content and the effect on the strength of fabric determined. In the usual procedure the fabric is buried for a set period (Anon., 1942; Bertolet, 1944; United States Army, 1944) and then removed and the tensile strength determined, though Batson *et al.* (1944) considered it preferable to determine the time taken for the fabric to lose 85 per cent. of its strength, and a similar view is expressed by Dean *et al.* (1945).

Soil composted according to ordinary greenhouse practice has been used in all our tests. This soil has a high organic content, contains micro-organisms active in digesting cellulose, and has the texture of a sandy loam.

To determine the optimum moisture content for soil burial tests, using this soil, 12-oz. cotton duck strips were buried in soil at 40, 50, 60, 70, 80, and 90 per cent. of the water-holding capacity, and four strips from each removed after 4, 7, 11, and 14 days, and the tensile strength determined. The results are shown in Table 3 and are presented graphically in Fig. 5.

These results indicated that, when the moisture content was 50-70 per cent. of the W.H.C., there was little variation in loss of tensile strength, but with soil of 80 per cent. W.H.C. deterioration was more marked. The rate of loss of tensile strength at 90 per cent. W.H.C. was high during the first seven days, but then fell off rapidly. This effect is probably explained by the poor aeration of soil approaching saturation with water. Growth of micro-organisms (apart from

anaerobic bacteria) would therefore tend to decline because of accumulation of inhibiting materials, such as carbon dioxide, and because of depletion of oxygen from the soil.
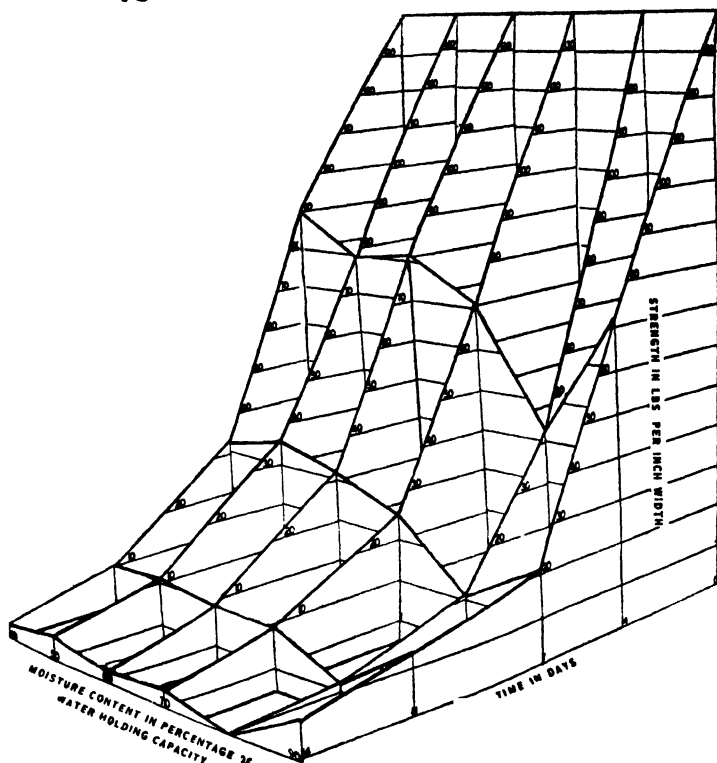
FIG. 5.—Effect of soil moisture content on rate of loss of strength of tensile strength of untreated cotton duck.

For routine tests, a soil moistened to 65-70 per cent. of its W.H.C. was used since it was found that if soil was moistened to the point when it felt moist when squeezed between the fingers, but would still crumble if dropped from a height of 1 foot on to the bench, it contained that amount of moisture. This rough test saved much time in determining moisture content accurately by drying and weighing the soil, and as the experiment quoted above demonstrates, a slight error in that region has little effect on the results.

The method adopted was to place a layer of the prepared soil of correct moisture content, 1 inch deep in the bottom of refrigerator boxes. The soil was compacted by tapping the box several times. The unsterilized fabric was pressed on to the soil, and a layer of soil half an inch deep placed above the strip and compacted. The refrigerator boxes were incubated at 30°C. for fourteen days, being weighed twice a week during incubation, and water added by spraying to replace any loss by evaporation. After removal from the soil, strips were washed with water, sterilized with methylated spirits, and dried; circles were washed and dried. The relevant physical tests were then carried out.

TABLE 3.—EFFECT OF SOIL MOISTURE CONTENT ON RATE OF LOSS OF TENSILE STRENGTH

Number of Days Burial	Percentage Moisture Content											
	40		50		60		70		80		90	
	Log T.S.	T.S.	Log T.S.	T.S.	Log T.S.	T.S.	Log T.S.	T.S.	Log T.S.	T.S.	Log T.S.	T.S.
4	1·98	91	1·91	81	1·91	81	1·86	72	1·04	44	1·83	68
7	1·53	34	1·59	39	1·52	33	1·44	28	0·98	9·5	1·34	22
10	1·08	12	1·12	13	1·03	11	1·01	10	0·44	3	1·19	16
14	0·71	5	0·82	7	0·65	7	0·69	5	0·00	1	0·94	9
General Means for Moisture Content	1·32	21	1·36	23	1·28	19	1·25	18	0·77	6	1·32	21

Since the range of the original figures was proportional to the means, they were converted to logarithms for analysis (Cochran, 1938). The value cited for tensile strength (T.S. lb. per inch width) in the table is the antilogarithm of the mean of the logarithm. The analysis of variance as described by Snedecor (1940) was applied with the following results:—

Difference for significance between general means of moisture content at 5 per cent. probability level (expressed as logarithms) .. .. 0·10

Difference for significance between general means of moisture content at 1 per cent. probability level (expressed as logarithms) .. .. 0·13

Difference for significance between sub-class means (individual entries in table) at 5 per cent. probability level (expressed as logarithms) .. .. 0·20

Difference for significance between sub class means (individual entries in table) at 1 per cent. probability level (expressed as logarithms) 0 26

Overseas procedures demand that fabric be immersed to a depth of 5 inches, probably to ensure constant moisture content and temperature. It was considered that this precaution was only necessary in tests under greenhouse conditions, or when the moisture content was not controlled. The use of a shallow depth of soil allows better aeration of the soil, and is more economical of space.

4. Discussion

There is considerable difference of opinion as to the value of the various laboratory methods of assessing the resistance to attack by micro-organisms. This is discussed by Barker *et al.* (1943).

Soil burial seems the logical method for testing such materials as sandbags and cordage, which would normally come into contact with soil during service. Some workers, however, consider that soil burial or soil suspension methods are of more significance than pure culture tests for all materials. From results obtained here, it seems probable that most materials which withstand soil burial would also withstand pure culture tests, though cases have been noted when the loss of waterproofness in the *Aspergillus niger* test was greater than in soil burial tests. However, soil burial would be likely to eliminate treatments that would give good results on materials which do not normally come into contact with soil. A similar view has been expressed by Dean *et al.* (1945). In service, tentage materials do, on occasions, become contaminated with mud, but it is doubtful whether soil burial is comparable to such contamination. The soil suspension method developed by Furry and Zametkin (1943) would appear to approximate more closely to such conditions. Although some workers have found difficulty in reproducing results in soil burial tests, we have found it fairly satisfactory in this respect, though close agreement between different laboratories cannot perhaps be expected.

One of the strongest objections to the pure culture tests is that fungi more tolerant to fungicides than the test organisms may be encountered in the field. For this reason, workers should not restrict their attention to one or two fungi.

Whatever type of test is used, some physical determination such as loss of tensile strength or waterproofness should be used as the criterion. The amount of visible growth, which has formed the basis of some specifications, may be entirely misleading, and it has been found that vigorous growth of fungi is not always accompanied by loss of waterproofness, nor is the absence of macroscopically visible growth always an indication that waterproofness has not been reduced.

It is probable that the failure to obtain complete correlation between laboratory tests and performance in the field is due in large measure to inadequate conditioning treatments. Our laboratory washing tests, while apparently severe, did not cause the loss of as much mercury as mild weathering. Washing tests described in overseas specifications and literature generally consist of 24 hours' washing in water at 30°C. In our tests a longer washing time was employed, though the temperature was lower. However, tests of this nature are suspect since they do not reproduce the alternate wetting and drying which occurs in service.

' For testing new materials it would therefore be desirable to expose treated pieces to natural weathering before submitting them to laboratory tests. Exposure under tropical conditions would be preferable, though valuable results may also be obtained under temperate conditions.

5. Acknowledgments

The mycological work described was conducted at the Plant Research Laboratories of the Victorian Department of Agriculture.

The physical determinations were made at the Munitions Supply Laboratories, Maribyrnong. The work described formed part of a cooperative investigation conducted by the Department of Munitions and the Victorian Department of Agriculture, and much of the work has been described in a hitherto restricted report (Cox *et al.*, 1945).

Mr. J. E. Cummins, who was Director of the Scientific Liaison Bureau at the time the work was conducted, assisted by providing some of the equipment for mycological testing, made overseas reports on current work available, and showed active interest in the progress of the work.

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Effect of Some Micro-organisms on the Physical Properties of Cotton Duck

By G. C. Wade, M.Agr.Sc.*

Summary

The effects of a number of fungi on the tensile strength and water-proofness of treated and untreated cotton duck are compared.

Metarrhizium sp. was the most active species tested, in reducing tensile strength of untreated duck. *Memnoniella echinata, Stachybotrys atra,* and *Chaetomium globosum* were also shown to be very strong cellulose digesters.

Various *Aspergillus* and *Penicillium* species were shown to reduce the waterproofness of the cotton duck.

1. Introduction

During the war period, numerous specimens of tentage which exhibited either loss of strength or waterproofness were received from the New Guinea area. These samples were examined for the presence of moulds and a considerable number of species isolated. Those species isolated frequently from deteriorated materials, or those related to species known to cause loss of tensile strength or waterproofness, were tested for those properties. Certain of the fungi which were very active in reducing tensile strength were compared carefully to assess their value for laboratory tests on rotproofness of cotton fabrics.

2. Effect on Loss of Tensile Strength

(i) *Method*

The tests were conducted using an accelerating medium essentially as described by Thom *et al.* (1934) and with the formula:—

Sodium nitrate	.. .. ..	3·0 g.
Potassium phosphate	.. ..	1·0 g.
Potassium chloride	.. ..	0·25 g.
Magnesium sulphate	.. ..	0·25 g.
Agar	.. ..	10·0 g.
Water	.. ..	1,000 ml.

A layer of the medium about ¼ inch deep was poured into the bottom of refrigerator boxes. Strips of cotton duck each 2½ inches wide and 11 inches long, cut in the direction of the warp, were suspended above the agar by folding over glass rods, which were prevented from slipping by a small piece of rubber, and the boxes then autoclaved.

After autoclaving, the cotton threads holding the strips were cut and the strips pushed down in uniform contact with the agar, for a length of four inches (see Wade, 1947b, Figs. 1 and 2). The work was conducted in a transfer chamber sterilized with propylene glycol aerosol (Wade, 1947a).

* Now an officer of the Tasmanian Department of Agriculture; the work described was carried out while the author was on the staff of the Victorian Department of Agriculture and a member of the Mycological Panel of the Scientific Liaison Bureau.

The strips were then inoculated by spraying, by means of a de Vilbiss atomizer, with a spore suspension of the organism tested. The boxes were incubated at a temperature of 28–30°C. for fourteen days, and the tensile strength determined.

(ii) *Results*

The results of tests with a number of organisms are shown in Table 1. The figures given are the average of three or more readings. As the tests were not all conducted at the same time, the figures are not strictly comparable, but only give an indication of the activity of the organisms.

TABLE 1

Organism	Effect on Appearance of Duck	Percentage Loss in Strength
Actinomyces sp.	Cream discolouration of duck. Strong earthy odour produced	80
Aspergillus niger	Black growth over surface of duck	Nil
Bacterium (Isolate A)	No discolouration	50
Bacterium (Isolate B)	No discolouration	70
Chaetomium sp. (probably *C. globosum*)	Yellow discolouration of duck. Abundant dark-grey perithecia formed on surface	95
C. globosum (culture from U.S.A.)	Yellow discolouration of duck. Abundant dark-grey perithecia formed on surface	95–100
Chaetomium sp. (isolated from sandbag)	Yellow discolouration of duck. Abundant dark-grey perithecia formed on surface	80
Cladosporium sp.	Greenish-black discolouration of duck. No surface growth	30–50
Curvularia (*Brachysporium*) sp.	Brown discolouration of duck. Little surface growth	85
Fusarium sp. (*Roseum* section)	White surface growth	25
Hormodendron sp.	Reddish-purple discolouration of duck. Slight white and brown surface growth	85
Memnoniella echinata	Copious black mould growth over surface of duck	100
Metarrhizium sp. (culture from U.S.A.)	Black moist growth over surface of duck	100
Penicillium sp. (sclerote producing form)	No observable effect	Nil
Penicillium luteum	Green surface growth. Slight orange discolouration of duck	Nil
Pestalozzia sp.	Grey discolouration of duck. Black spore masses on surface of duck	30–50
Pullularia pullulans	No observable effect	5–15
Stachybotrys atra	Copious dull-black growth over surface of duck	100
Sterile fungus—*Rhizoctonia* type	Dark-grey discolouration of duck. No surface growth	80–95
Trichoderma sp.	Fabric not discoloured. White surface growth with olive green spore masses	15

Curvularia (*Brachysporium*) sp. and the sterile fungus were very frequently isolated from tentage from the New Guinea area, and the brownish or grey irregular patches caused by them were the most

common type of damage noted on samples obtained from the area. However, as the results show, they were less active in reducing strength of cotton duck than several other fungi, particularly *Memnoniella echinata*, *Stachybotrys atra*, and certain of the *Chaetomium* isolates. Their growth rate was slower than those latter species, and they produced spores less abundantly, so that it was difficult to obtain uniform results following inoculation with them. Therefore they were not used in routine rotproofing tests.

Of the very active cellulose-digesters, *Memnoniella echinata* was isolated most frequently. It was particularly common on tentage from very humid situations. *Stachybotrys atra* was not common on tentage from New Guinea but was commonly isolated from deteriorated cellulosic materials from the Melbourne area.

Hormodendron sp. was isolated from reddish purple areas on duck. Such areas were fairly frequent on duck from similar situations to those showing infection with *Curvularia* sp. and the sterile fungus, and it was similar in its capacity to digest cellulose.
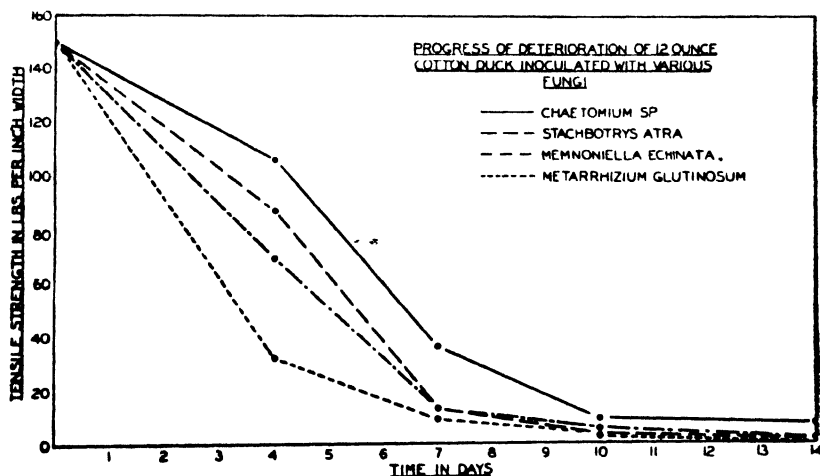
FIG. 1.—Progress of deterioration of 12-oz. cotton duck inoculated with various fungi (the curve for *Chaetomium globosum* has been omitted because there was no significant difference between the effect of this fungus and *Memnoniella echinata*).

Pestalozzia sp. was almost universally present on tentage from New Guinea, but as the results show, it is not a particularly active cellulose digester.

Pullularia pullulans was isolated particularly readily from samples of tentage, which had been treated by the copper soap—hot wax method, and which had shown extreme loss of strength after use in New Guinea. However, inoculation of duck with *P. pullulans* resulted in little growth, and it seems likely that the loss of strength of the samples from which it was isolated was primarily due to light-tendering. It is therefore not known why *Pullularia* was so consistently associated with this type of breakdown.

Thaysen and Bunker (1927) give descriptions of many of these fungi and a bibliography of micro-organisms attacking cellulose.

The data quoted in Table 1 only give an approximate indication of the relative activity of the fungi in digesting cellulose. In order to determine this more accurately for five of the most active forms, a further test was conducted in a similar manner, but four strips were removed after incubation for 4, 7, 10, and 14 days. The growth of the organisms was stopped immediately by immersing the strips in methylated spirits, and the tensile strengths were then determined.

The results are shown in Table 2, and illustrated graphically in Fig. 1.

Metarrhizium sp. caused more rapid deterioration than the other fungi tested. This result is consistent with the work of Greathouse *et al.* (1942). Deterioration was least rapid with *Chaetomium* sp. and there was no significant difference between the effects of *S. atra, M. echinata,* and *C. globosum.*

3. Relative Effect of Fungi on Tensile Strength of Rotproofed Cotton Duck

Tests were conducted to determine the relative effects of *M. echinata* and *S. atra* on duck treated by various processes. In these tests the fabric was sterilized by methyl alcohol vapour as described in a previous paper (Wade, 1947b). The results are shown in Table 3. There was no significant difference in the activity of *M. echinata* and *S. atra* on the copper treated fabrics tested, except in the case of unwashed caustic soda modified copper treated ducks, when *M. echinata* was more active. It is of interest to note that Jensen (1946) found that *M. echinata* is slightly more copper tolerant than *S. atra.* However, *M. echinata* caused significantly greater loss of strength of duck, finished to the American specification J.Q.D. 242, than did *S. atra.*

4. Effect of Various Fungi on Waterproofness of Cotton Duck

It has been known for a considerable time that a number of common moulds, particularly *Aspergillus* spp. and *Penicillium* spp. are capable of growing on cotton duck (Hardy, 1942; Thaysen and Bunker, 1927). The presence of these fungi was considered objectionable for aesthetic reasons, but their possible effect on other properties of the duck has apparently not been studied previously.

During the work conducted here, it was noted than these fungi grew vigorously on both untreated and copper soap treated cotton duck. Dr. H. L. Jensen, Macleay Bacteriologist, Linnean Society of New South Wales, independently noted this fact, and work first carried out by Dr. Jensen and the Munitions Supply Laboratories showed that, *Aspergillus niger* was capable of causing loss of waterproofness of copper soap treated fabric (Jensen, 1946).

Tests were then conducted to determine the effect of a number of fungi on the waterproofness of untreated cotton duck.

(i) *Method*

Four-inch squares of untreated 18-oz. cotton duck were suspended above water in glass jars. The jars were then sterilized by autoclaving, and the duck inoculated by spraying with spore suspensions

TABLE

Number of Days Incubation	Metarrhizium sp.		Stachybotrys atra		Memnoniella echinata		Chaetomium globosum		Chaetomium sp.	
	Log. T.S.	T.S.	Log. T.S.	T.S.	Log. T.S.	T.S	Log. T.S.	T.S.	Log. T.S.	T.S.
4	1·49	31	1·94	87	1·84	69	1·79	62	2·03	107
7	0·94	9	1·13	13	1·13	17	1·19	16	1·66	36
10	0·54	3	0·59	4	0·71	6	0·79	6	0·99	10
14	0·27	2	0·23	2	0·29	2	0·29	2	0·89	8
General Means for Fungi	0·81	6·5	0·97	9	1·01	10	1·01	10	1·37	23

Since the range of the original figures was proportional to the means, they were converted to logarithms for analysis. The value cited for tensile strength (T.S., lb. per in. width) in the table is the antilogarithm of the means of the logarithms. The analysis of variance technique described by Snedecor was applied in analysing the results.

Difference for significance between general means for fungi at 5 per cent. probability level (as logarithms) = 0·09.

Difference for significance between general means for fungi at 1 per cent. probability level (as logarithms) = 0·12.

Difference for significance between sub-class means (individual entries in table) at 5 per cent. probability level (expressed as logarithms) = 0·18.

Difference for significance between sub-class means (individual entries in table) at 1 per cent. probability level (expressed as logarithms) = 0·24.

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TABLE 3.

Nature of Fabric	Treatment	Tensile Strength, lb. per in. Width		
		Before Inoculation	After Inoculation and Incubation with *S. atra*	After Inoculation and Incubation with *M. echinata*
12 oz. duck	Copper soap-wax emulsion	137	104	80*
12 oz. duck	Copper soap double bath—			
	As received	162	174	170*
	After washing 7 days in a water spray	167	139	133*
18 oz. duck	Copper soap double bath (caustic soda modified)—			
	As received	170	180	128†
	After washing 7 days in a water spray	186	179	180†
12 oz. duck	Copper tannate (factory process)	176	90	84*
18 oz. duck	Phenyl mercuric acetate	192	199	206*
12 oz. duck	Material nominally finished to specification J.Q.B. 242	193	201	105‡

* No significant difference between effects of the two fungi.

† Difference for significance at 5 per cent probability level 7 and at 1 per cent. level 11.

‡ Difference for significance at 5 per cent probability level 30 and at 1 per cent. level 45.

of a number of fungi. After incubation for 14 days, the waterproofness, in terms of the head of water required to produce leakage, was determined at the Munitions Supply Laboratories. The experiment was conducted in quadruplicate.

(ii) *Results*

The results are shown in Table 4.

TABLE 4.—EFFECT OF VARIOUS FUNGI ON THE WATERPROOFNESS OF 18-OZ. DUCK

Organism	Waterproofness	Remarks
Aspergillus niger	½	Vigorous black surface growth
A. penicilloides	½	Vigorous green surface growth
A. ustus	2	Vigorous buff surface growth
A. glaucus	1	Moderate green surface growth
A. flavus	2	Vigorous yellow-green surface growth
A. sydowi .	8	No obvious growth
Penicillium luteum	3½	Vigorous green surface growth
Pestalozzia sp.	5	Grey stained irregular areas
Memnoniella echinata	7	Vigorous black surface growth
Cladosporium sp.	9	Greenish-black diamond shaped areas. No surface growth
Uninoculated	10	

Difference for significance at 5 per cent. probability level $= 1\cdot5$.
Difference for significance at 1 per cent. probability level $= 2\cdot0$.

Aspergillus niger, A. penicilloides, A. ustus, A. glaucus, and *A. flavus* caused extensive loss of waterproofness. Although *Cladosporium* sp. and *Memnoniella echinata* grew vigorously on the duck, they did not cause marked loss of waterproofness.

The reason for the effect of these fungi on waterproofness has not been definitely established, but it is probable that they destroy the oils and waxy materials normally present on the surface of the fibres. It is suggestive that the most active fungi are members of the *Aspergillus* and *Penicillium* genera, which have the ability to break down fats and oils (Horowitz-Wlassowa and Liwschitz, 1935), and that Eyre (1932) showed that *A. niger* and *A. flavus* were more powerful in breaking down fats and oils than *A. sydowi*.

5. Effect of Fungi on Waterproofness of Rotproofed Cotton Duck

(i) *Method*

Four-inch circles of the treated duck were sterilized by exposure to methyl alcohol vapour, and then rested on the mineral salts and agar medium described earlier in this report. They were then inoculated by spraying with spore suspensions of the fungi. After incubation for 14 days at 30°C., the waterproofness was determined.

(ii) *Results.*

The results are shown in Table 5 and demonstrate that both *Aspergillus ustus* and the *Penicillium* sp. are more mercury tolerant than *A. niger. A. ustus* appears from these results to be slightly more copper sensitive than *A. niger* or *Penicillium* sp.

TABLE 5.

Nature of Fabric	Treatment	Waterproofness, Inches, after Inoculation with—		
		A. niger	*A. ustus*	*Penicillium* sp.
18-oz. duck ..	Phenyl mercuric acetate (0 017 per cent. mercury on fabric)—			
	As received	8 (n.s.)	0 (m)	8 (v.sl.)
	After washing	6 (v.sl.)	0 (v)	½ (v.)
12-oz. duck ..	Copper soap double bath (soda ash modified)—			
	As received	½ (v.sl.)	3 (v.sl.)	½ (sl.)
	After washing	0 (m)	0 (m.)*	½ (v.)

* Copper soap showed moderate decolourisation

Results are the means of four determinations—

n.s. = No apparent growth.
v.sl. = Very slight growth.
m = Moderate growth.
v. = Vigorous growth.

6. Acknowledgments

The work described was conducted at the Plant Research Laboratories, Department of Agriculture, Victoria. As with a previous paper on a related subject, it was part of a cooperative investigation with the Munitions Supply Laboratories, Maribyrnong, Victoria. Messrs. A. B. Cox, W. R. Hindson, and K. N. Mortensen of those Laboratories were closely associated with the work, which was in part described in a hitherto restricted report (Cox *et al.*, 1945).

Mr. J. E. Cummins, former Director of the Scientific Liaison Bureau, assisted with the work in many ways, particularly by making available overseas reports written during the war period and by his active interest in the work.

Miss G. E. McQuienn and Miss N. M. Fenwick-Barbour, formerly of the staff of the Munitions Supply Laboratories, assisted capably with the laboratory work.

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Fusarium Wilt of Tomato in Australia

2. Inheritance of Field Immunity to Fusarium Wilt in the Tomato (*Lycopersicon esculentum*)

By E. M. Hutton, M.Sc.,* Margaret Mills, B.Sc.,* and J. E. Giles†

Summary

It has been shown that Pan America is a suitable parent for introducing field immunity to Fusarium wilt into new tomato hybrids suited to Australian conditions. The use of Pan America in some crosses leads to an improvement in vitamin C content.

Simple crosses have resulted in the incorporation of field immunity in suitable hybrids but multiple crosses containing Pan America in two places are recommended.

The inheritance mechanism for field immunity, although a simple one, does not follow the usual course, and appears to be influenced by both the female and pollen parent used.

1. Introduction

Previous work (Mills and Hutton, 1946) suggested that strains of *Fusarium bulbigenum* var. *lycopersici* of varying pathogenicity existed in Australia. It was demonstrated that *Lycopersicon pimpinellifolium* and the variety Pan America derived from it by Porte and Walker (1941) are highly resistant to all the Australian isolates of the organism tested. This high degree of resistance to the development of the organism results in field immunity to Fusarium wilt in Pan America.

Inheritance of immunity to Fusarium wilt in hybrid progenies has been studied by Bohn and Tucker (1940) who found that immunity depended on a single dominant genetic factor. From crosses they developed hybrids homozygous for immunity and having good agronomic quality. Porte and Walker (1941) using similar methods produced Pan America.

In Australia, Pan America, although producing good quality fruit, is a poor yielder under most conditions, and has not been generally accepted by growers. The work summarized in this paper shows that the field immunity to Fusarium wilt of Pan America is heritable, and that by using it as a parent, field immune hybrids suitable to Australian conditions can be developed by plant breeders.

2. Materials and Methods

Inoculation methods were similar to those used previously (Mills and Hutton, 1946). Five-weeks-old tomato seedlings were inoculated by dipping the roots in a water suspension of macerated mycelium and spores of a highly pathogenic strain of *Fusarium bulbigenum* var. *lycopersici* grown on a modified Tochnai medium. The seedlings were planted in pots in the greenhouse, and 28 days after inoculation were rated on the extent of vascular browning (internal rating), as this method had been shown less liable to error than rating based on the outward appearance of the plant. Average internal ratings of Bonny Best and Pan America checks used throughout the experiment were 8·1 and 0·76 respectively. Table 1 gives a comparison between some of the Bonny Best and Pan America checks.

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TABLE 1.—INTERNAL RATINGS OF FUSARIUM INOCULATED BONNY BEST
AND PAN AMERICA CHECKS

Variety	Number of Plants rated Internally as—											Total	Percentage Rated 0–2	Percentage Rated 3–10
	0	1	2	3	4	5	6	7	8	9	10			
Bonny Best ..	1	5	8	8	14	8	27	24	50	64	130	339	4·1	95·9
Pan America ..	184	180	17	3	4	..	2	7	..	..	..	397	96·0	4·0

0–2 Vascular browning absent or confined to root system.
3–10 Vascular browning extending upwards in the stem.

In the majority of Bonny Best plants vascular browning extended towards the tops. These plants and hybrids of similar susceptibility were rated internally from 3–10 and were classed susceptible to Fusarium wilt. Vascular browning did not extend above ground level in most Pan America plants which were thus rated 0–2 as shown in Table 1. Pan America and plants in hybrid progenies given this rating do not wilt if left growing in the greenhouse and so are classed as having field immunity to Fusarium wilt.

A number of crosses using the field immune Pan America were made at Canberra. For comparison, an F4 selection of a cross between *L. pimpinellifolium* and Marglobe received as 42–50–6 from Dr. B. L. Wade of United States of America, has been used as the field immune parent in other crosses. The susceptible varieties used in the Canberra crosses varied in resistance to Fusarium wilt and were grouped previously (Mills and Hutton, 1946).

3. Inheritance of Field Immunity to Fusarium Wilt in the Progeny of Crosses

(i) *Crosses between Susceptible Parents*

TABLE 2.—NUMBERS OF FIELD IMMUNE AND SUSCEPTIBLE PLANTS IN THE
PROGENY OF CROSSES BETWEEN SUSCEPTIBLE PARENTS

Variety	No. of Lines	Genera-tion	Field Immune	Sus-ceptible	Total
Rouge de Marmande (R.D.M.) x Pearson 29–17	..	F2	38	215	253
Rouge de Marmande (R.D.M.) x Pearson 29–17	5	F3	31	169	200
Rouge de Marmande (R.D.M.) x Pearson 29–17	2	F4	1	44	45
Vetomold (V.) x Rouge de Marmande (R.D.M.)	1	F3	..	19	19
Pearson x Earliana	6	F3	4	114	118
Bounty x 926–142–1 (Wade) ..	7	F3	10	283	293
Tatinter x 926–142–1 (Wade) ..	11	F3	4	434	438
Tatura Dwarf Globe x 926–142–1 (Wade)	10	F3	11	407	418
(V. x R.D.M.) x Riverside	3	F3	..	53	53
(V. x R.D.M.) x Pearson	6	F3	..	106	106
(R.D.M. x Pearson 29–17) x R.D.M.	..	F1	..	89	89
			99	1,933	2,032

Table 2 shows that the majority of plants in hybrid progenies from crosses between susceptible parents are field susceptible. Most of the field immune segregates are in the Pearson 29–17 cross, indicating that if sufficient hybrid work were done with this less susceptible parent, field immune hybrids of good agronomic quality might be produced. It is considered that a large proportion of the field immune plants in Table 2 come within the experimental error, since 4 per cent. of Bonny Best plants are classed as field immune in Table 1.

(ii) *Crosses between a Field Immune and Susceptible Parent*

The results in Table 3 show that in the first generation field immunity is completely dominant over susceptibility.

TABLE 3.—NUMBERS OF RESISTANT AND SUSCEPTIBLE PLANTS IN THE F1 OF CROSSES BETWEEN SUSCEPTIBLE PARENTS AND THE FIELD IMMUNE PARENTS PAN AMERICA AND 42–50–6 (Wade)

Variety	Genera-tion	Field Immune	Sus-ceptible	Total
Bounty x Pan America (P.A.) ..	F1	40	..	40
Tatinter x P.A.	F1	20	..	20
Bounty x 42–50–6	F1	81	..	81
Tatinter x 42–50–6 . ..	F1	88	1	89
		229	1	230

Tables 4 and 5 show the numbers of field immune and field susceptible segregates in the F2 and more advanced progenies of crosses between the susceptible varieties Rouge de Marmande, Bounty, Tatinter, and Tatura Dwarf Globe, and the field immune male parents Pan America and 42–50–6 (Wade). The F2 progenies, were tested in the greenhouse without field selection. All other progenies, with the exception of one, resulted from agronomic selections in the field. The exception is the F3 progeny marked G.H. in the Rouge de Marmande x Pan America cross. This resulted from F2 plants not given an internal rating and which did not succumb to Fusarium wilt in the greenhouse.

TABLE 4.—NUMBERS OF RESISTANT AND SUSCEPTIBLE PLANTS IN THE PROGENY OF CROSSES BETWEEN A FEMALE SUSCEPTIBLE PARENT AND THE FIELD IMMUNE MALE PARENT PAN AMERICA

Variety	No. of Lines	Genera-tion	Field Immune	Sus-ceptible	Total
R.D.M. x Pan America (P.A.) ..	..	F2	438	57	495
R.D.M. x Pan America (P.A.) ..	17	F3	564	104	668
R.D.M. x Pan America (P.A.) ..	20 G.H.	F3	323	28	351
R.D.M. x Pan America (P.A.) ..	5	F4	454	68	522
R.D.M. x Pan America (P.A.) ..	5	F5	321	34	355
Bounty x P.A.	..	F2	79	17	96
	1	F3	1	42	43
Tatinter x P.A.	..	F2	405	29	434
	8	F3	277	74	351
Tatura Dwarf Globe x P.A. ..	..	F2	317	38	355
	2	F3	59	19	78
			3,238	510	3,748

The important points illustrated by Tables 4 and 5 are the preponderance of field immune plants in most progenies and the high proportion of agronomic field selections with immunity to Fusarium wilt. The greater than 3 : 1 ratios of field immune to field susceptible plants in the F2's are difficult to explain on the basis of the operation of a single dominant gene as shown by Bohn and Tucker (1940). A simple type of inheritance must be involved since Table 3 shows dominance to be complete in the F1. Bohn and Tucker (1940) noted a number of cases with a higher proportion of immune progeny than expected when the Fusarium immune parent was the male. To explain this they considered the factor for immunity to be linked with a factor influencing the effectiveness of the microgamete. It is possible that the greater than 3 : 1 ratios in Tables 4 and 5 could be explained on this basis as in all instances the field immune parents Pan America and 42–50–6 were used as the male. No reciprocal crosses of those in Tables 4 and 5 were made so that the hypothesis of Bohn and Tucker (1940) could not be fully tested. The preponderance of the field immune factor is more marked when 42–50–6 (Wade) is used as the pollen parent. There is some indication that different female susceptible parents may influence the number of field immune progeny produced in crosses with field immune male parents, so that modifying factors may be operating.

TABLE 5.—NUMBERS OF RESISTANT AND SUSCEPTIBLE PLANTS IN THE PROGENY OF CROSSES BETWEEN A FEMALE SUSCEPTIBLE PARENT AND THE FIELD IMMUNE MALE PARENT 42–50–6 (Wade)

Variety	No. of Lines	Generation	Field Immune	Susceptible	Total
Bounty x 42–50–6	..	F2	346	2	348
	3	F3	89	42	131
Tatinter x 42–50–6	..	F2	373	15	388
	3	F3	127	4	131
Tatura Dwarf Globe x 42–50–6	..	F2	469	39	508
	4	F3	143	40	183
			1,547	142	1,689

The excess of resistant types makes the problem of combining agronomic suitability and field immunity to Fusarium wilt easier. In the Rouge de Marmande-Pan America cross of Table 4, the F2 had a preponderance of field immune plants, so that a high proportion of the seventeen F3 lines field selected for agronomic quality had the immune character. Inoculation in the greenhouse showed that eight lines were homozygous for field immunity, seven heterozygous for field immunity, one heterozygous susceptible, and one homozygous susceptible. Of the twenty F3 lines selected from the F2 in the greenhouse for field immunity, nine were homozygous and eleven heterozygous for this character. On this basis it does not appear advantageous to select the F2 in the greenhouse for field immunity to Fusarium wilt. The inheritance pattern of field immunity to Fusarium wilt is maintained throughout the different generations of the Rouge de Marmande-Pan America cross and the other crosses of Tables 4 and 5. That its basis is sound is shown by the fact that field immune hybrids evolved by this technique have remained free from Fusarium wilt under conditions of widespread field infection of susceptible varieties.

(iii) *Multiple Crosses Including One or Two Field Immune Parents*

Table 6 gives the numbers of field immune and susceptible plants occurring in the progenies of multiple crosses containing one and two field immune parents respectively. The second cross containing both 42–50–6 (Wade) and Pan America gave the greatest numbers of resistant parents. Multiple crosses like the first in Table 6 are of little practical use as it would be difficult to select the desired combination of agronomic quality and field immunity to Fusarium wilt from the progenies.

TABLE 6.—NUMBERS OF FIELD IMMUNE AND SUSCEPTIBLE PLANTS IN MULTIPLE CROSSES INVOLVING ONE OR TWO RESISTANT PARENTS

Variety	Genera-tion	Field Immune	Sus-ceptible	Total
(V. x R.D.M. x Pearson 29–17) x (V. x R.D.M. x P.A.)	F2	25	473	498
(42–50 x V. x R.D.M.) x (V. x R.D.M. x P.A.)	F2	438	105	543
		463	578	1,041

4. Field Performance of Fusarium Wilt Immune Hybrids Developed from the Crosses

Table 7 gives the field performance in the C.S.I.R. plots at Red Cliffs, Victoria, of some of the advanced hybrids showing field immunity to Fusarium wilt. These hybrids were developed from the crosses shown in Tables 4, 5, and 6. In the trial plots 16 plants of each variety or hybrid were randomized in each of six blocks so that 96 plants of each were transplanted. During the 1946-47 season Fusarium wilt was prevalent so that the yielding ability in lb. per plant in Table 7 reflects to some extent the resistance to Fusarium wilt of the various varieties and hybrids. Pearson 29–17 has been the outstanding variety in replicated trials at Red Cliffs over a number of years and is taken as the standard by which new varieties or hybrids are evaluated. ‘

Of the advanced hybrids in Table 7, 7A–2–1, 12A–1–1, 12A–6–1, and 12A–8–1 yielded more heavily than Pearson 29–17, none of the differences being statistically significant. On the basis of first grade fruit over 2½ in. in diameter, 7A–2–1, 12A–6–1, and 12A–8–1 gave higher yields than Pearson 29–17, the differences lacking significance as before. The hybrids 5A–3–1, 5A–4–1, and 12A–7–1 were still segregating a number of Fusarium wilt susceptible plants, whereas the other hybrids were almost fixed for field immunity although showing segregation for agronomic type.

In Table 7, Pearson 29–17 is among the lowest in vitamin C content. This is in accord with previous results from replicated trials carried out for several years at Red Cliffs. In these, Pearson 29–17, Rouge de Marmande, and Adelaide Dwarf Red have given average vitamin C contents from 35–38 mg. per 100 g., whereas Pan America, Grosse Lisse, and Riverside have ranged from 45 to 48 mg. per 100 g. Pan America

has usually been the best variety for vitamin C. The results in Table 7 indicate that the high vitamin C content of Pan America is heritable, since the last four hybrids, particularly 12A-7-1 and 12A-8-1, are among the best in this respect. At the second harvest all the advanced hybrids had significantly greater vitamin C contents than Pearson 29-17.

TABLE 7.—YIELD AND VITAMIN C CONTENT OF FUSARIUM WILT IMMUNE HYBRIDS IN COMPARISON WITH STANDARD VARIETIES AT RED CLIFFS, 1946-47 SEASON

Variety	Cross	Weight of Fruit lb./plant			Average Vitamin C Content in mg./100 g.	
		Fruit over 2¼ in. diam.	Fruit less than 2¼ in. diam.	Total	1st Harvest	2nd Harvest
Adelaide Dwarf Red	.. .. ..	2·7	1·1	4·9	41·75	39·57
Pearson 29-17 ..	.. .. ..	4·4	3·1	9·1	38·07	30·49
Riverside	.. .. ..	3·0	1·5	6·4	45·08	47·39
Red Cloud ..	.. .. ..	3·1	1·2	6·3	43·90	40·25
5A-3-1	Rouge de Marmande x 42–50 (Wade)	0·5	3·9	7·7	35·65	43·25
5A-4-1	Rouge de Marmande x 42–50 (Wade)	0·6	2·5	5·2	35·34	45·54
7A-2-1	(Vetomold x R.D.M.) x Pan America	4·8	3·2	9·9	27·19	43·64
10A-4-1	(Vetomold x R.D.M.) x 42–50–6 (Wade)	0·3	3·6	8·1	39·76	45·34
12A-1-1	R.D.M. x Pan America ..	1·7	5·8	9·7	40·14	46·46
12A-6-1	R.D.M. x Pan America ..	5·0	3·4	11·0	43·29	42·27
12A-7-1	R.D.M. x Pan America ..	3·5	1·5	6·8	54·33	46·79
12A-8-1	R.D.M. x Pan America ..	4·8	3·0	10·4	50·08	46·41

In yield min. diff. for significance at 5 per cent. point = 2·8 lb.
In vitamin C min. diff. for significance at 5 per cent. point = 8·04 mg.

5. Discussion

For plant breeders, the important point shown by this paper is the relative ease with which the combination of field immunity to Fusarium wilt and good agronomic quality can be achieved by the use in crosses of the easily available variety Pan America. It appears that with its use hybrids with yields as good as standard varieties can be developed. There is also the possibility of an improvement in vitamin C content with Pan America hybrids.

The preponderance of field immune segregates in the crosses described is a puzzling feature of the work. Until further experiments are done it is not possible to understand fully the mechanism involved. Until proved otherwise, it is best to use Pan America as the pollen parent. The work described gives an indication of the results that can be expected with different types of crosses. The backcross method has not been used as it is considered that, with the possible exception of Pearson 29–17, most of the standard varieties need agronomic improvement as well as improvement in disease resistance qualities.

Although the simple cross involving a susceptible and field immune parent has been largely used for the purpose of this paper it is possibly not the best type of cross for the plant breeder to use. Multiple crosses as shown in Table 6 including four or more varieties have a greater chance of improving the tomato agronomically as well as introducing field immunity to Fusarium wilt. It is well known that many of the desirable agronomic characters are dependent on the operation of multiple factors, so that improvements in agronomic type are more likely to result from multiple crosses containing several varieties with the desirable characters. Where multiple crosses are used, it is apparent that to introduce field immunity to Fusarium wilt in a sufficient proportion of segregates, Pan America should occur in at least two places.

Where field immunity to Fusarium wilt is being introduced into the segregates of crosses, agronomic field selection should be followed by a test of field immunity to the disease by a greenhouse technique along the lines described. It is inadvisable to rely on selection for immunity to the disease under conditions of field infection, as the development of Fusarium wilt in plants growing in the field is greatly influenced by environmental conditions.

6. Acknowledgments

Messrs. R. D. Brock and R. R. Rochford of the Division of Plant Industry were responsible for the data obtained in the later stages of this work. Mr. J. Webster of the Council's Commonwealth Research Station, Merebin, kindly made available his vitamin C figures for the hybrids and varieties in the Red Cliffs trials. Mr. G. McIntyre statistically analysed the data from this trial.

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The Sub-surface Atmosphere of Wheat Infested with *Rhizopertha dominica* F.

By R. F. Powning, A.S.T.C.*

Summary

The results of some studies on the composition of the sub-surface atmosphere and insect distribution in Victorian bulk-wheat depots are presented, together with data from laboratory experiments.

In laboratory studies, the carbon dioxide concentration reached 15 to 20 per cent. when an infestation of *Rhizopertha dominica* F. was started either at the surface or deep in the wheat contained in tall narrow jars. A surface infestation distributed itself evenly through the wheat and produced a large number of second generation adults, but any insects leaving the vicinity of the wheat surface were killed. The insects in a deep infestation showed no tendency to migrate away from the high carbon dioxide concentration in the bottom of the jar, some surviving an atmosphere of between 10 per cent. and 18 per cent. carbon dioxide for eight weeks—but there was an almost complete absence of second generation adults.

Analyses of the intergranular atmosphere of stored bulk-wheat did not reveal gases other than the normal constituents of air. Carbon dioxide increased owing to respiration processes within the wheat bulk, and the oxygen varied in a complementary manner. In the heavily infested wheat, the carbon dioxide concentration rose to about 2·5 per cent. in the vicinity of the maximum insect population (consisting mainly of *Rhizopertha dominica* F.). The temperature of both infested and uninfested wheat varied in the same manner as the carbon dioxide.

1. Introduction

The restriction of the insect infestation to the periphery of bulk-wheat has been described by Wilson (1945a), and it has been shown that the phenomenon may be satisfactorily explained by the presence of unsuitable temperature-humidity conditions beneath the wheat surface (Wilson, 1945a, 1947; Birch, 1945). However, before the true explanation had been demonstrated, it was thought that the apparently uninhabitable zone in the wheat might be due to toxic constituents in the intergranular air. Abnormal carbon dioxide or oxygen concentrations were considered likely to be contributing factors, and the data obtained in analyses of the sub-surface atmosphere of stored bulk-wheat are presented in this paper.

The laboratory experiments described in the next section were undertaken to determine, under somewhat simplified conditions, the nature and extent of the changes in the intergranular atmosphere caused by a developing insect population, and the effect of these changes on that population.

2. Laboratory Studies

(i) *Experimental Technique*

Rhizopertha dominica F., being the most serious pest in bulk-wheat in Victoria, was chosen for these laboratory experiments, which were planned as complementary studies to the field experiments. The test insects were 0-7 days old and reared in the

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laboratory under controlled conditions. The experiments were carried out at 90°F. (32°C.) and 65 per cent. R.H. in cylindrical glass jars, 5 ft. tall by 3 in. internal diameter, filled to the top with conditioned wheat and covered with fine wire gauze lids. To facilitate the insect counts, discs of wire gauze, of sufficiently large gauge to permit free movement of the insects, were inserted at one foot intervals in the jars as they were filled; lengths of ¼-in. glass tubing, embedded in the wheat to the appropriate depth, served for drawing the gas samples. Infestations were initiated by the addition of 500 adult insects to either the top or the bottom of the jars. In both series of experiments observations were made on separate jars after periods of 3, 7, and 10 days, and 2, 4, 6, and 8 weeks, the population count terminating each experiment.

A Haldane gas analysis apparatus was used for the carbon dioxide analyses, samples being taken in duplicate and their mean used for the graphs. Each foot of wheat was examined separately for live and dead insects, the results being recorded as histogram blocks in the illustrations.

(ii) *Carbon Dioxide Bulid-up in Insect-free Wheat*

As a check on carbon dioxide production in wheat by micro-organisms or by the respiration of the wheat itself, two jars were set up with wheat of low moisture content (9·24 per cent.) and two with wheat of relatively high moisture content (11·4 per cent.). One of each of these was hermetically sealed, the other left open, and the intergranular air analysed for carbon dioxide after a four-week period at 90°F. (32°C). The sealed relatively moist wheat produced a concentration of 2·4 per cent. carbon dioxide, but both of the jars containing dry wheat and the unsealed jar with the moist wheat developed less than 0·5 per cent.

This is in agreement with figures quoted by Dendy and Elkington (1920) who showed, when using a smaller quantity of a dry wheat under slightly different conditions, that the concentration of carbon dioxide after seven days was 0·48 per cent.

(iii) *Behaviour of a Surface Infestation*

When placed on the surface, the insects showed a strong tendency to move downwards, and in a few days live insects were found in all depths of the wheat. After about four weeks, there was little further insect movement and a high mortality occurred together with the high carbon dioxide concentration which had reached 15-20 per cent.

When it was found that anomalous carbon dioxide figures, not comparable with the smooth trends shown in the other experiments, were revealed in the four-weeks experiments, this was repeated (four weeks B). The histograms show that, although the total insect population and its distribution was much the same as in the original four-weeks jar, only those insects which remained near the surface survived. The only explanation that can be offered for this difference is that, for some reason, breeding in the first jar is subnormal and irregular, whereas in the duplicate jar normal numbers of late-stage larvae were present, which might be expected to raise the carbon dioxide concentration markedly. Howe and Oxley (1944) have shown that fourth instar larvae of *Calandra* produce more than three times as much carbon dioxide as any other stage, and it is possible that a similar relation exists in *Rhizopertha*.
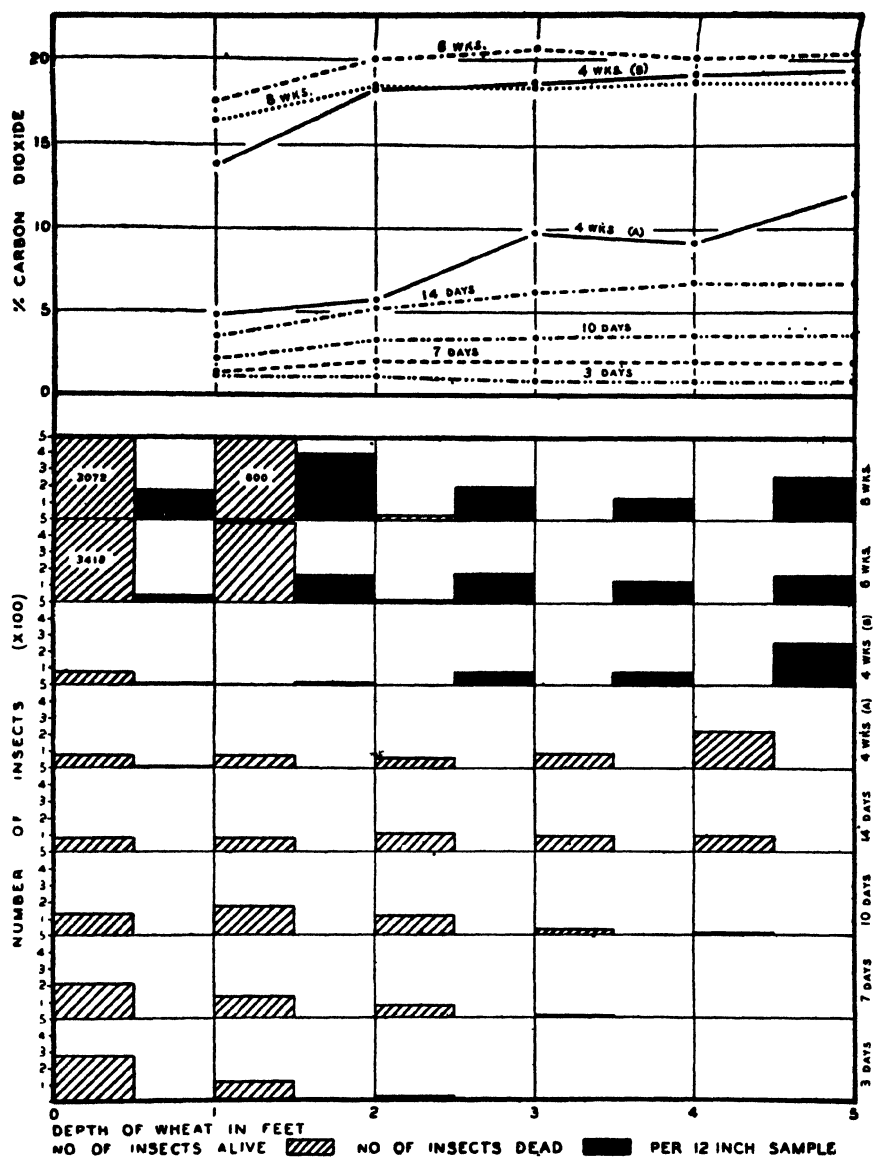
FIG. 1.—Changes in carbon dioxide concentration and insect distribution in tall glass jars when infested at the surface with *Rhizopertha dominica* F.

(iv) *Behaviour of a Deep Infestation*

In the deep infestation there was an immediate increase in carbon dioxide concentration in the lower parts of the jar, but the insects showed no tendency to migrate up through the wheat away from it. All the insects lived in an atmosphere of 10-15 per cent. carbon dioxide for 11 days, and some were alive after 8 weeks in an atmosphere of 10-18 per cent. carbon dioxide.

Contrary to the results of the surface infestation, very little breeding occurred under these conditions, the counts indicating an absence of second generation adults at six weeks; in the eight-weeks count the increase was only about 30 per cent.

3. Studies in Bulk-wheat Depots

(1) *Experimental Technique*

The war-time bulk-wheat storage depots in Victoria consisted of two huge sheds, each about 1,000 ft long by 200 ft. wide, in which the wheat, to the extent of several million bushels, formed a continuous heap, retained by bulkheads of heavy timber. Infestation
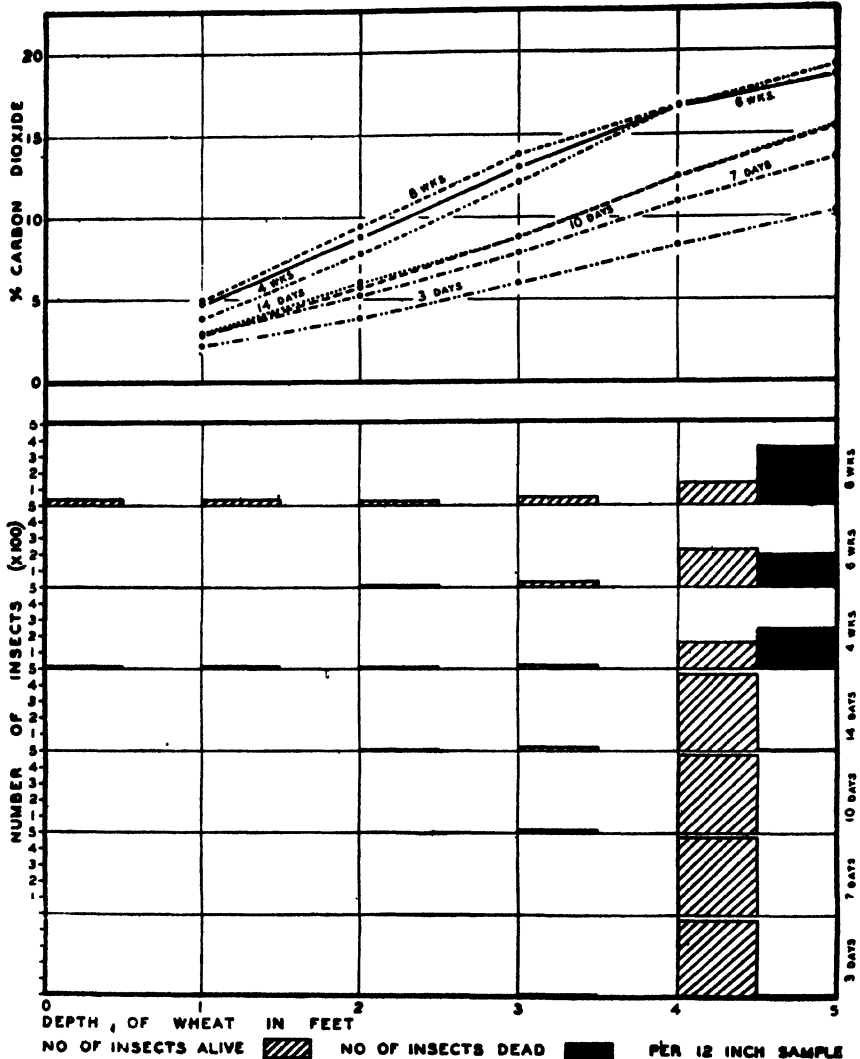
Fig. 2.—Changes in carbon dioxide concentration and insect distribution in tall glass jars when infested at the bottom with *Rhizopertha dominica* F.

consisted mainly of *Rhizopertha dominica* F., but in certain situations, considerable numbers of *Latheticus oryzae* Waterh. and small numbers of other insects were present (Wilson, 1945*b*).

In selecting sites for the collection of the analytical data, areas that had been recently fumigated or that were close to a bulkhead or the crest were avoided. The gas samples were taken at the centre of an infested area with the aid of a levelling bulb and suitable lengths of ½-in. diameter copper tubing. The analyses were carried out with a Sleigh portable gas analysis apparatus.

Temperatures at various depths in the wheat were determined by thermometers attached to wooden probes. A special metal probe of 1-in. internal diameter, subdivided into 6-in. sections, was used for sampling the grain at different depths and determining the distribution of the insect population. The numbers of live and dead insects separated from the samples so obtained are included in the form of histograms in Figs. 3 and 4.

(ii) *Results of Observations and Analyses*

Typical sets of figures are presented to illustrate the variations in carbon dioxide, oxygen, temperature, and insect numbers with depth of wheat. They were obtained from:—

> (*a*) Insect-free wheat, 1942-43 season, "No. 6 Pool" (15-18 months in storage); and 1943-44 season, "No. 7 Pool" (3-6 months in storage).
>
> (*b*) A light infestation. 1941-42 season (27-30 months in storage).
>
> (*c*) A medium infestation. 1941-42 season (27-30 months in storage).
>
> (*d*) A heavy infestation. 1941-42 season (27-30 months in storage).

In both lots of insect-free wheat (Fig. 3*a*) the temperature and the carbon dioxide rose gradually from a low value near the surface to become constant below about 3 feet, but, while the curves were similar, the 1943-44 wheat was slightly cooler and had a lower carbon dioxide concentration.

With the appearance of a light infestation (Fig. 3*b*) the character of both the carbon dioxide and the temperature curves changed markedly. Insects were found throughout the whole depth sampled, but most of them were concentrated within 18 inches of the surface. This distribution was reflected in an increase in the percentage of carbon dioxide and the temperature, the oxygen concentration showing a corresponding decrease.

In Figs. 4*a* and 4*b* are presented data from a medium and a heavy infestation respectively. The insects were more numerous, though fewer at depths below two feet than in the light infestation. The graphs of carbon dioxide and temperature show much higher values, and the oxygen concentration is lower, than in Fig. 1*b*, but the curves are essentially similar, i.e., the maxima and minima occur at a depth in the wheat corresponding to the position of highest insect population.

FIG. 3.—(a) Variation in carbon dioxide concentration and temperature with depth in cool uninfested wheat.

(b) Variation in carbon dioxide and oxygen concentration, temperature and insect numbers in a light infestation.

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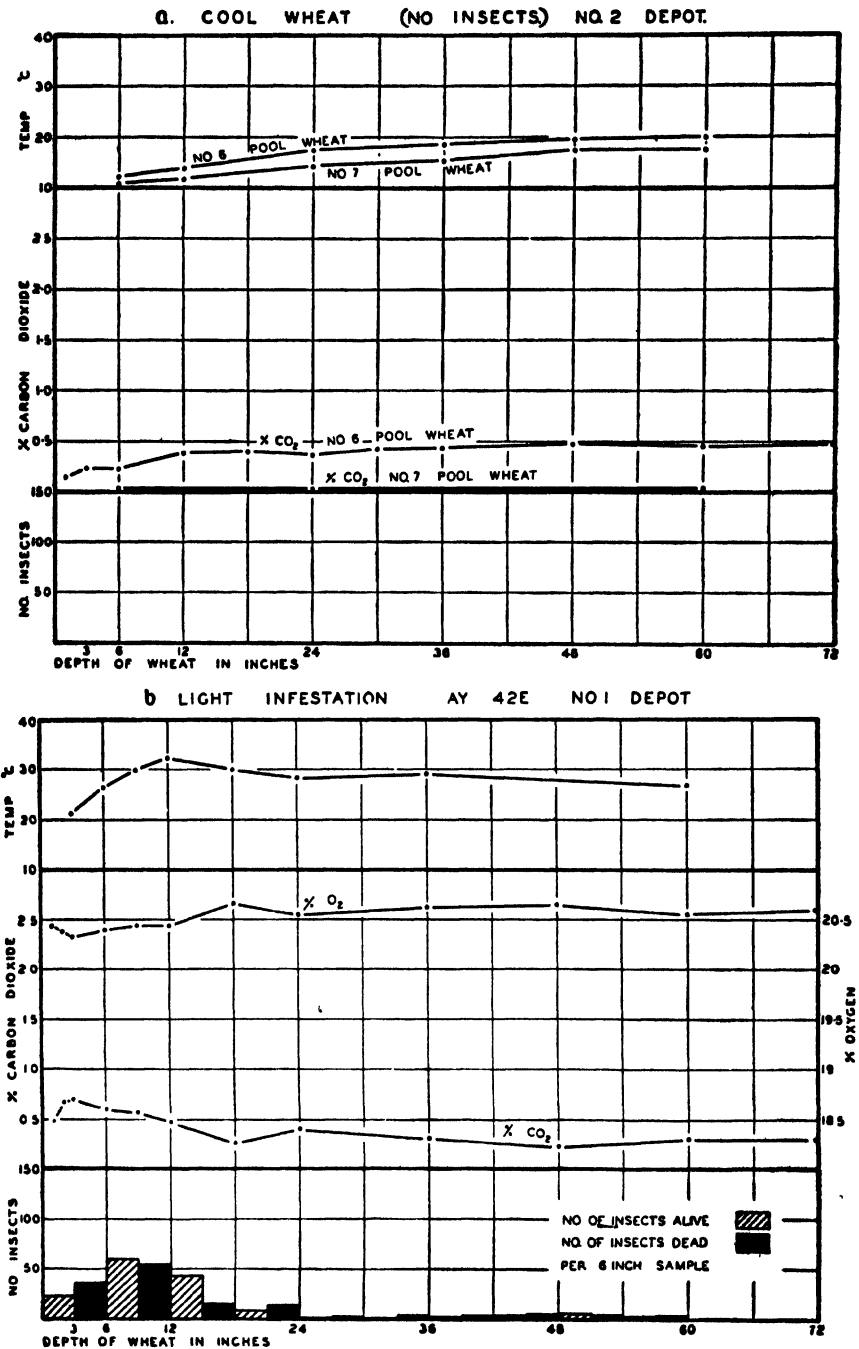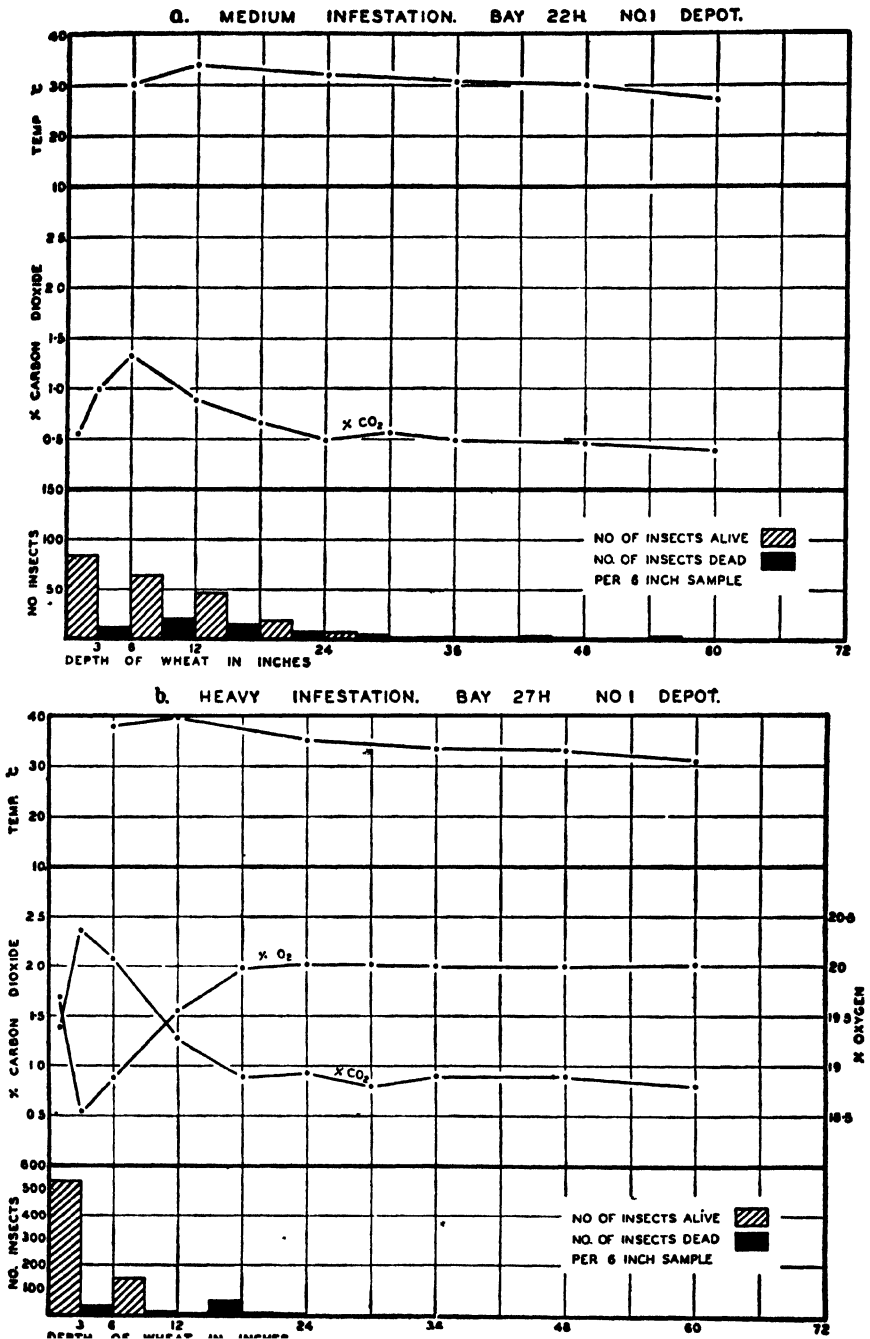
FIG. 4.—(a) Variation in temperature, carbon dioxide concentration, and insect numbers in wheat with a medium infestation.
(b) Variation in temperature, carbon dioxide and oxygen concentrations, and insect numbers in heavily infested wheat.

4. Discussion

It is reported that in Western Australian depots where infestations generally are much more severe than in the Victorian depots, carbon dioxide concentrations of about 5 per cent. occurred (Wilson, 1945c). Oxley and Howe (1944) made many determinations of carbon dioxide in intergranular air of infested grain, and found that the concentration was normally less than 1 per cent. They also state, however, that where the infestation is heavy, and a considerable bulk of wheat is heating, concentrations of 4-5 per cent. may occur. In Victoria, a large number of analyses were carried out on samples from wheat under various conditions of infestation, but on no occasion was the carbon dioxide concentration higher than 3 per cent., or the oxygen figure more than 3 per cent. below normal. Analyses for carbon monoxide, aldehydes, and hydrocarbons showed that these were not present in the intergranular atmosphere.

The tolerance of *Rhizopertha* to high carbon dioxide concentrations (5-10 per cent.) and the rather surprising absence of any reaction on the part of the insects to a gradient of carbon dioxide concentration, confirm the validity of Wilson's conclusion that the limitation of insect infestations to the periphery of bulk wheat is adequately explained by temperature and humidity conditions that develop within the grain mass.

5. Acknowledgments

The author wishes to thank Mr. F. Wilson and Mr. F. J. Gay for their willing advice and cooperation.

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The Addition of Skim Milk Powder to Australian Bread

By R. C. Hutchinson,* D.Sc., H. E. West,† B.S.A., and Jeanette T. Jeffreys*

Summary

Typical Australian skim milk powders were incorporated in test doughs on a 6 per cent. dry matter basis. The resultant loaves were immature, the volumes poor, the crusts harsh, and the crumbs coarse, open, and with frequent cores. Unsuccessful attempts were made to eliminate these defects by subjecting the milk to various treatments and by using flour from harder wheats.

Skim milk powders that were known to have excellent baking qualities under United States conditions were also incorporated in test doughs where they were found to have the same depressing effect on the resultant loaves as the Australian powders.

In Australia it is not customary to add cane sugar or fat to the common loaf but, in the United States, both these constituents are used. Baking tests were therefore conducted on doughs containing each and both of these constituents. The results of these tests showed that, when small amounts of fat are added to doughs containing skim milk powder, the resultant loaves are greatly improved. Furthermore, it was found that a loaf, superior in all respects to the water-loaf control, could be obtained by adding the fat in the form of an emulsion. Several fats were tested.

Test bakes were also conducted on doughs containing skim milk powder, fat, and small amounts of several surface-modifying ingredients and on doughs containing various German whole egg and egg albumen substitutes made from skim milk. The loaves resulting from all these bakes were inferior to those containing skim milk powder and fat in the form of an emulsion.

As a result of baking tests with skim milk powders containing several fats in different percentages, it was concluded that satisfactory results could be obtained with a powder containing 20 per cent. fat and that the most desirable amount of powder to add to bread was 12½ per cent.

The results of several large scale baking tests in which skim milk powder, containing 20 per cent. mutton fat, was used on 12½ per cent. dry matter basis are briefly described. In general, these results confirmed those obtained in the laboratory.

1. Introduction

There is no article of food so generally and universally consumed as bread. In many countries it is the basic food of a large majority of the people and, if this bread is low in any important dietary constituent, the health of the people will almost certainly suffer in consequence. Unfortunately, ordinary white bread is a poor source of several essential food constituents which include the amino acids, lysine and tryptophane; calcium; and the vitamins riboflavin and thiamin. On the other hand, skim milk powder is a particularly good source of all these constituents and, by incorporating skim milk solids in bread, a loaf with a much higher nutritive value results.

In the United States, it is common practice to add 6 per cent. skim milk powder to bread, although rye bread only contains 4-5 per cent., whilst the standard school lunch loaf contains 12 per cent. and special high calcium loaves may contain as much as 40 per cent.

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In Australia, it is not customary to add skim milk powder to the common loaf, the only breads containing milk powders being fancy breads for which there is a limited demand. When skim milk powder is incorporated in the common Australian loaf, there is an appreciable reduction in volume, the crust is harsh and immature, and the crumb coarse, open, and with frequent cores. This is contrary to the results obtained in the United States where it is claimed that the addition of skim milk powder to bread improves every physical characteristic of the loaf.

The object of this investigation was to determine the reason for the detrimental effect which skim milk powder has on the physical properties of Australian bread and to determine whether this detrimental effect can be eliminated without losing any of the enhanced nutritive value of the bread or increasing appreciably its cost.

2. Experimental Test Bakes

(i) *Experimental Formula, Technique, and Evaluation*

During the investigation, many test bakes were conducted, and the formula for the control doughs, the baking technique, and the method of evaluating the finished loaves, are described hereunder.

(a) *Formula.*—The following formula was used:—

Flour	..	..	300 g. = 100 per cent.
Salt	..	..	6 g. = 2 per cent.
Compressed yeast	..	5·4 g. = 1·8 per cent.
Improver	..	..	1 g. = 0·33 per cent.
Water	..	..variable = 58-60 per cent.

(b) *Technique.*—The yeast was first shaken with 30 cc. of water until a fine, homogeneous suspension was formed and the salt and improver dissolved in a further 144 cc. of water which had been heated to the temperature necessary to bring the temperature of the dough, after the mixing of all ingredients, to 26·6°C. (80°F.). All the ingredients were then mixed for two minutes in a laboratory model Hobart mixer during which time additional water was added if necessary. The dough was then placed in a jar in a fermentation cabinet held at 26·6°C. The jar was part of a closed system which permitted the measurement of the rate and degree of fermentation by the displacement of a saturated salt solution. When the displacement had reached 1,100 cc., and without breaking the closed system, the jar was knocked to assist the release of more gas and fermentation then allowed to continue until the total volume of gas evolved was 1,400 cc. Then 400 grams of dough were taken, " handed up," replaced in the fermentation cabinet for 10 minutes' recovery, moulded into a square mould, placed in a baking tin, and returned to the fermentation cabinet for proofing. During the proof period, the formation of a skin was prevented by keeping the atmosphere moist with steam. The dough was then placed in an oven at 221°C. (430°F.) for 25-30 minutes.

(c) *Evaluation.*—Sixteen hours after they were baked, all test loaves were scored in the following manner.

A water-loaf control, which was included in each batch baked, was used as a standard for comparison and given an arbitrary value of 100. Points, based on the following properties, were then allotted to each loaf:—(*a*) volume, (*b*) maturity, (*c*) crust characteristics, and (*d*) crumb characteristics. No definite values were ascribed to aroma or palatability although a constant watch was kept for abnormalities.

(ii) *Skim Milk Powder in Ordinary Australian Bread*

Six typical Australian skim milk powders, all of which had been bought on the open market, were incorporated in test doughs to the extent of 6 per cent. on a dry matter basis. The use of 6 per cent. skim milk powder provided milk solids equivalent to those which would have been provided had the entire liquid content of the dough been fresh skim milk. The powders were added dry and, with the exception of an increased fermentation time, there were no modifications in the baking technique. The results of these tests are given in Table 1. Fig. 1 is a diagrammatic representation of a section of the control loaf and of sections of two test loaves, one containing spray-dried and the other roller-dried powder. Both the loaves containing skim milk powder had similar physical properties.

TABLE 1.—THE EFFECT OF ADDING SKIM MILK POWDER TO AUSTRALIAN BREAD

Reference Number	Powder	Bread Score
Control		100
1	Spray-dried	55
2	Spray-dried	50
3	Spray-dried	45
4	Roller-dried	45
5	Roller-dried	45
6	Roller-dried	45

From Table 1 it will be seen that the addition of skim milk powder, either spray-dried or roller-dried, produced a very inferior loaf. The volumes were poor, the control loaf having a volume of 1,420 cc. and the test loaves only 1,220 cc. The test loaves were also very immature, the crusts harsh, and the crumbs, coarse, open, and with frequent cores.

WATER LOAF CONTROL   LOAF CONTAINING SPRAY-DRIED POWDER   LOAF CONTAINING ROLLER-DRIED POWDER
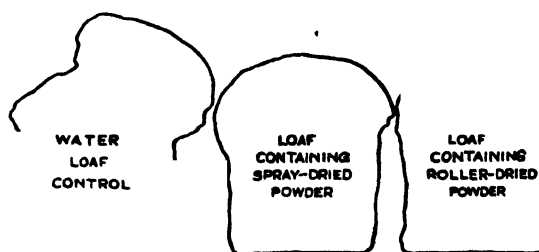
FIG. 1.

In further tests, the batter or ferment method of mixing doughs was employed. In this method a batter or ferment consisting of—

Flour	30	g.
Milk powder	18	g.
Improver	1	g.
Water at 49°C.	20	cc.
Yeast solution at 32°C.	34·5	cc.

was allowed to ferment for one hour after which it was mixed in the usual way with the remaining flour, salt, and liquor. The dough was then fermented to 1,200-1,300 cc. of gas. The results of these tests are given in Table 2. From this table it will be seen that the batter or ferment method of mixing doughs containing skim milk powder was not as satisfactory as the method employed previously. The loaves were extremely immature and the volumes very poor.

TABLE 2.—THE ADDITION OF SKIM MILK POWDER TO BREAD PREPARED BY THE BATTER METHOD

Reference Number	Powder	Bread Score
Control	.. ..	100
7	Spray-dried	45
8	Roller-dried	40

(iii) *Preheated Skim Milk Powders*

The results of experiments conducted by Greenbank *et al.* (1927) indicated that the preheat treatment to which skim milk is subjected before being dried affects its baking qualities; powder which has received a relatively high heat treatment yields a loaf of greater volume and better texture than powder which has been subjected to a less severe heat treatment. The following year Grewe and Holm (1928) conducted more detailed baking tests in which they used three different flours, and spray-dried skim milk powders that had been preheated to 50°, 63°, 73°, 83°, 93°, and 100°C. for 30 minutes. With all flours, the poorest results were obtained in bread containing powder which had been subjected to a preheat treatment of 50°C. A preheat treament of 63°C. gave a slightly better result but there was a marked improvement in the loaves containing powders that had been preheated to 73°, 83°, 93°, and 100°C. Skovholt and Bailey (1931) found that preheat treatments at 77°, 88°, and 96°C. for 30 minutes greatly improved the baking qualities of skim milk powders and that the improvement was approximately the same for each treatment. In later experiments, Ashworth *et al.* (1942) used spray-dried powders which had been preheated at 82-88°C. (180-190°F.) for 30 minutes, and recently Harland, Ashworth, and Golding (1943) used powders which had been preheated to 82°C. for 45 minutes or 90-95°C. for 5 minutes.

Hence, it would appear that in the baking of bread the most satisfactory results are obtained from skim milk powders which have been preheated to a temperature of approximately 85°C. for 30 minutes or to a higher temperature for a shorter period.

During the manufacture of roller-dried powder, milk is first heated to a temperature of approximately 100·5°C. for about 10 minutes and then to approximately 120°C. for about 3 seconds. When milk is spray-dried, the heat treatment is less severe and depends upon the method of drying and the frequency with which the powder is removed from the drying chamber. In most Australian factories the temperature of the milk does not exceed 80°C. and the powder may not remain at this temperature for more than a few minutes.

In order to obtain samples which were known to have received adequate heat treatment, arrangements were made for the manufacture of four samples of powder from milk which had been specially preheated to 85°C. for 30 minutes. Three of the samples were spray-dried, the temperature of the drying chamber being 79-80°C., and the fourth was roller-dried at a steam pressure of 60 pounds per square inch. The first sample, which was from the first of the milk to be sprayed into the drying chamber at the beginning of the day's run, had remained in the chamber for approximately 14 hours before being collected. The second sample, which was from milk which had been sprayed into the drying chamber half way through the day's run, had remained in the chamber for approximately 7 hours before being collected. The third sample, which was from the last of the milk to be sprayed into the drying chamber before closing down for the day, had remained in the chamber for only a few minutes. When reconstituted all these powders had an acidity of 0·15 per cent. when expressed as lactic acid.

These powders were added to test doughs to the extent of 6 per cent. on a dry matter basis. They were added both wet and dry. When added wet, all liquor except that substracted for the yeast solution, was mixed with the milk powder and added to the flour in the form of a liquid. The results of the test bakes are given in Table 3. From this table, it will be seen that the additional preheat treatment did not improve the baking qualities of the powders. All the test loaves were very immature, the volumes poor, the crusts harsh, and the crumbs coarse, open, and with frequent cores.

TABLE 3.—THE ADDITION OF SPECIALLY PREHEATED SKIM MILK
POWDERS TO BREAD

Reference Number			Powder				Bread Score	
							Addition Wet	Addition Dry
Control	..	..	..	. .	..	..	100	
9	..	..	..	Spray, 14 hrs.	..	..	50	..
10	..	..	..	Spray, 14 hrs.	..	..	..	50
11	..	..	..	Spray, 7 hrs.	..	..	50	..
12	..	..	..	Spray, 7 hrs.	..	..	..	50
13	..	..	..	Spray, sev. mins.	..	..	45	..
14	..	..	..	Spray, sev. mins.	..	..	..	45
15	..	..	..	Roller	..	..	..	45	..
16	..	..	..	Roller	..	..	..	45

A sample of the second spray-dried powder was then reconstituted and portions of the reconstituted milk subjected to additional and more drastic heat treatments. These reconstituted milks were then added to test doughs so that each dough contained 6 per cent. skim milk solids on a dry matter basis. The results of the test bakes are given in Table 4. From these results it is evident that the additional heat treatment to which the reconstituted powders were subjected was not of great value in improving their baking qualities. There was only a slight improvement in volume and maturity.

TABLE 4.—THE ADDITION TO BREAD OF POWDERS SUBJECTED TO ADDITIONAL HEAT TREATMENTS

Reference Number			Additional Treatment				Bread Score
Control	..	..	..	..	..	..	100
17	..	..	90° C. for 5 minutes	..	.		55
18	..	..	95° C. for 5 minutes	..	..		55
19	..	..	100° C. for 5 minutes	..	..		50
20	..	..	110° C. for 5 minutes	..	..		65
21	..	..	90° C. for 15 minutes	..	..		55
22	..	..	95° C. for 15 minutes	..	..		50
23	..	..	100° C. for 15 minutes	..	..		55
24	..	..	90° C. for 30 minutes	..	..		50
25	..	..	95° C. for 30 minutes	..	..		55
26	..	..	100° C. for 30 minutes	..	..		55

When skim milk was heated to temperatures higher than 110°C. for five minutes, or slightly less, the colour was so dark that it adversely affected the colour of the bread.

(iv) *Acidified Fresh Skim Milks*

The doughs containing skim milk had a pH of approximately 5·8 whilst the controls had a pH of approximately 5·2. This suggested that the acidity of the skim milk may be an important factor determining its baking quality.

Samples of fresh skim milk, which had been heated to temperatures of 100° and 110°C., and held at these temperatures for approximately five minutes, were acidified by the addition of various amounts of lactic acid and used in the preparation of eight test doughs. On a dry matter basis these doughs each contained 6 per cent. skim milk solids. The results of the test bakes are given in Table 5. From the results of this table, it will be seen that an increased acidity greatly improved the baking qualities of fresh skim milk when heated to temperatures of 100° or 110°C. Also, at the lower acidities, milk heated to 110°C. had better baking qualities than milk heated to 100°C. In loaves 19, 27, and 29 the volumes were poor, in loaves 20, 28, and 31 they were fair, in loaf 30 fair to good, and in loaf 32 good. Loaf 19 was very immature, loaves 20 and 27 only slightly immature, and the remaining loaves of average maturity. In loaves 19-29, the texture was open and slightly coarse but in the remaining loaves it was fine and even. In all loaves, the colour of the crumb was good but the colour of the crust rather high.

Although an increased acidity, together with a comparatively severe preheat treatment, improved greatly the baking qualities of fresh skim milk, the resultant loaves were still inferior to an ordinary water loaf.

TABLE 5.—THE ADDITION OF ACIDIFIED FRESH SKIM MILK TO BREAD

Reference Number	Milk Acidity	Bread Score	
		Heat Treatment at 100° C	Heat Treatment at 110° C
Control	% ..	100	
19	0·17	50	..
20	0·17	..	65
27	0·31	60	..
28	0·31	..	70
29	0·39	65	..
30	0·39	.	85
31	0·50	85	..
32	0·50	..	85

(v) *Flours from Soft and Hard Wheats*

In all the foregoing bakes, a blend of average Victorian flours was used. These flours were milled mostly from soft wheats and, in order to investigate the effect of using a hard wheat flour, a blend containing 80 per cent. of hard, Queensland wheat flour was used. The results of bakes containing this flour are given in Table 6. In both loaves, the volumes were poor, the loaves slightly immature, and the crumbs open.

TABLE 6.—BREAD FROM HARD WHEAT FLOURS

Reference Number	Powder	Bread Score
Control	.. . . ..	100
33	Spray-dried . .	60
34 .	Roller-dried ..	50

(vi) *American Skim Milk Powders*

Samples of three skim milk powders, which were known to have excellent baking qualities under United States conditions, were next obtained from the American Dry Milk Institute in Chicago. Two of the samples had been spray-dried and one roller-dried. On a reconstituted basis, their acidities were 0·16, 0·16, and 0·17 per cent. respectively. These powders were added to doughs on a 6 per cent. basis, the doughs being made from the original blend of average Victorian flours. The results of the test bakes are given in Table 7.

From these results it is seen that, when added to Australian bread, American skim milk powders had the same depressing effect as Australian powders. The loaves were all very immature the volumes poor and the crumbs coarse, open, and with frequent cores.

TABLE 7.—THE ADDITION OF AMERICAN SKIM MILK POWDERS TO BREAD

Reference Number			Powder			Bread Score	
						Addition Wet	Addition Dry
Control	..	..	..	..	..	100	
35	..	..	Spray 1	..	..	50	..
36	..	..	Spray 1	..	..	..	50
37	..	..	Spray 2	..	..	50	..
38	..	..	Spray 2	..	..	..	50
39	..	..	Roller	..	..	50	..
40	..	..	Roller	..	..	..	50

(vii) *The Addition of Sugar and Fat to Bread*

The results of the preceding tests indicated that the depressing effect of skim milk powder on Australian bread was not due to any particular characteristic of the milk powder or the flour. It was suggested that it may be due to the fact that in Australia neither cane sugar nor fat is added to the common loaf whereas American breads contain both these ingredients. In order to investigate this point baking tests were conducted on doughs which contained (*a*) cane sugar plus additional fat, (*b*) cane sugar only, (*c*) additional fat only, and (*d*) neither cane sugar nor additional fat. On a dry matter basis, the cane sugar and additional fat were each added to the extent of 2 per cent. and the skim milk powder 6 per cent. The fat consisted of lard and all the additional ingredients were added dry. The results of the test bakes are given in Table 8.

TABLE 8.—THE ADDITION OF SKIM MILK POWDER, SUGAR, AND FAT TO BREAD

Reference Number			Powder			Sugar	Fat	Bread Score
Control	..	..	..	..	..	—	—	100
1	..	..	Spray	..	..	—	—	55
4	..	..	Roller	..	..	—	—	45
Control	..	..	..	..	..	+	—	100
41	..	..	Spray	..	..	+	—	60
42	..	..	Roller	..	..	+	—	45
43	..	..	Spray	..	..	+	—	55
44	..	..	Roller	..	..	+	—	45
Control	..	..	..	..	..	—	+	100
45	..	..	Spray	..	..	—	+	90
46	..	..	Roller	..	..	—	+	80
47	..	..	Spray	..	..	—	+	90
48	..	..	Roller	..	..	—	+	80
Control	..	..	..	..	..	+	+	100
49	..	..	Spray	..	..	+	+	90
50	..	..	Roller	..	..	+	+	80
51	..	..	Spray	..	..	+	+	90
52	..	..	Roller	..	..	+	+	80

From the results in Table 8, it will be seen that, although sugar hastens fermentation time, it is of little value in improving the physical quality of bread. In all the loaves containing sugar there were definite signs of immaturity, the volumes were poor and the texture open, coarse, and with numerous cores. On the other hand, the addition of fat improved the physical quality to a marked degree and, in all the loaves containing fat, there was an increase in volume, the loaves were of average maturity, and the textures even, although inclined to be " fuzzy."

(viii) *The Addition of a Fat Emulsion to Bread*

In the preceding bakes, the fat was mixed into the dough in the same way as the other ingredients. In the bakes which are about to be described, the fat was added in the form of an emulsion.

This emulsion contained the following constituents:—

Skim milk powder	..	180 g.
Sugar	.. ..	60 g.
Salt	.. ..	60 g.
Lard	.. ..	60 g.
Water	.. ..	360 g.

and each dough contained 72 grams. From the results presented in Table 9 and Fig. 2, it will be seen that, when lard is added in the form of an emulsion, the resultant loaves are greatly improved, being definitely superior to the control loaf. For both spray and roller dried powders, the loaves were of average maturity and the volumes good. In each loaf the texture was also good, although inclined to be slightly " fuzzy " in the loaf containing roller-dried powder.

TABLE 9.—THE ADDITION OF A LARD EMULSION TO BREAD

Reference Number	Powder	Bread Score
Control	.. ..	100
53	Spray-dried	115
54	Roller-dried	105
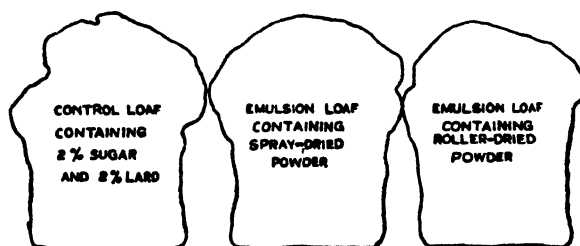
FIG. 2.

In order to investigate, in greater detail, the effect of adding fat to bread containing skim milk powder, tests were conducted in which lard was either directly mixed into the dough or added as an emulsion.

The method of adding the milk powders, which had been previously studied, was also varied, the addition being made dry, wet, or as part of the emulsion. The results are presented in Table 10. From these results, it is evident that there was no, or little, differences in breads containing lard that had been mixed into doughs to which skim milk powder had been added in the dry and wet forms. When the lard and milk powder were added in the form of an emulsion. however, there was a marked improvement. In the loaves containing milk powders in the wet and dry forms, there was slight immaturity, the volumes ranged from fair to good, and, in general, the crumbs were soft, fine, and even, although inclined to be slightly open in the first and last bakes. When lard and milk powder were added in the form of an emulsion, the loaves were of average maturity, there was a marked improvement in the volume, the crumb was soft, fine, and even, and only in the last bake was there any inclination to openess.

TABLE 10.—THE ADDITION OF LARD TO BREAD

Reference Number	Powder	Addition	Bread Score
Control	.. ..		100
55	American Spray, 1	Dry	90
56	American Spray, 1	Wet	90
57	American Spray, 1	Emulsion	110
58	American Spray, 2	Dry	90
59	American Spray, 2	Wet	90
60	American Spray, 2	Emulsion	110
61	American Roller	Dry	95
62	American Roller	Wet	95
63	American Roller	Emulsion	120
64	Spray, 14 hrs.	Dry	90
65	Spray, 14 hrs	Wet	90
66	Spray, 14 hrs	Emulsion	110
67	Spray, 7 hrs	Dry	90
68	Spray, 7 hrs	Wet	90
69	Spray, 7 hrs	Emulsion	115
70	Spray, sev. mins	Dry	80
71	Spray, sev. mins	Wet	80
72	Spray, sev. mins	Emulsion	110
73	Roller	Dry	80
74	Roller	Wet	80
75	Roller	Emulsion	105

So far lard had been the only fat used, but, in Australia, there are four other common edible fats or oils each of which deserves consideration. These are beef fat, mutton fat, peanut oil, and maize oil. The results of baking tests in which 2 per cent. of these fats have been added to doughs containing 6 per cent. spray-dried skim milk powder are given in Table 11. The plasticity of the hydrogenated oils was approximately equal to that of lard.

From the results in Table 11, it will be seen that for all the fats and hydrogenated oils, the best results were obtained with the fats in the form of an emulsion. The beef suet emulsion gave the best results although good results were obtained with beef fat, mutton

fat, mutton suet, and hydrogenated peanut and hydrogenated maize oil emulsions. It is interesting to note that neither unhydrogenated peanut nor maize oil gave improved results when added in the form of emulsions. In no loaf was there any noticeable difference in palatability or aroma due to the presence of the additional fat.

TABLE 11.—THE ADDITION OF VARIOUS FATS AND OILS TO BREAD

Reference Number	Fat or Oil	Addition	Bread Score
Control	..	.	100
76	Beef fat	Dry	95
77	Beef fat	Emulsion	110
78	Beef suet	Dry	100
79	Beef suet	Emulsion	115
80	Mutton fat	Dry	95
81	Mutton fat	Emulsion	105
82	Mutton suet	Dry	100
83	Mutton suet	Emulsion	110
84	Peanut oil	Dry	65
85	Peanut oil	Emulsion	60
86	Peanut oil hyd	Dry	80
87	Peanut oil hyd	Emulsion	110
88	Maize oil	Dry	65
89	Maize oil	Emulsion	60
90	Maize oil hyd	Dry	75
91	Maize oil hyd	Emulsion	105

In all the loaves, with the exception of those containing unhydrogenated peanut and maize oils, the volumes ranged from good to very good. Peanut and maize oils, however, had a depressing effect upon volume although the colour was quite good. The loaves containing beef and mutton fats and unhydrogenated peanut and maize oils had an open but even texture and numbers 84 and 88 contained occasional cores. In the other loaves, the texture was fine, even, and soft. Beef and mutton suets and hydrogenated peanut oil produced loaves of average maturity, whilst unhydrogenated peanut and maize oils produced loaves which were definitely immature. Numbers 77, 81, and 91 were also of average maturity, numbers 76, 80, and 90 being slightly immature.

(ix) *Interface Modifying Ingredients*

In a dough containing added fat, small amounts of lecithin, or glycerol monostearate, are known to have a modifying effect on the interfaces between the particles of fat and the other dough constituents. It was thought that by adding a small amount of one of these substances to a dough into which fat had been mixed this interface modification might be sufficiently pronounced to produce a loaf equal to that produced when fat is added in the form of an emulsion.

In order to investigate this point, twelve baking tests were conducted on doughs containing 6 per cent. spray-dried skim milk powder, 2 per cent. lard, and small amounts of interface modifying ingredients. These ingredients were mixed with water before their addition. The results of the bakes are given in Table 12.

From the results of Table 12, it will be seen that the only bake in which the resultant loaf was superior to the control loaf was that in which 0·2 per cent. glycerol monostearate was added. Nevertheless, this loaf was still inferior to a loaf containing the same amount of fat in the form of an emulsion, the principal defect being a slight immaturity of the crust.

Because superglycerinated shortenings are not available in Australia, experiments with these fats could not be conducted.

TABLE 12.—THE ADDITION OF FAT AND AN INTERFACE MODIFYING CONSTITUENT TO BREAD

Reference Number	Constituent	Amount	Bread Score
		%	
Control	.. ..	..	100
92	Egg lecithin	0·0002	95
93	Egg lecithin	0·002	95
94	Egg lecithin	0·02	90
95	Egg lecithin	0·2	90
96	Soya bean lecithin	0·0002	95
97	Soya bean lecithin	0·002	95
98	Soya bean lecithin	0·02	90
99	Soya bean lecithin	0·2	95
100	Glycerol monostearate ..	0·0002	90
101	Glycerol monostearate ..	0·002	95
102	Glycerol monostearate ..	0·02	100
103	Glycerol monostearate ..	0·2	105

(x) *Skim Milk Powders Prepared as Egg Substitutes.*

Scientific and technical missions, sent by the Commonwealth Government to Germany, have included in their reports references to several whole egg and egg albumen substitutes which have been used with good results by German bakers. The production of these substitutes was fostered by the German Government about 1934 in its drive to discourage the importation of goods which required foreign currency. Several of the most popular substitutes, which contain considerable quantities of skim milk, are described hereunder.

" Milei V " is a whole egg substitute made in the following manner: Into 300 litres of skim milk are dissolved 600 grams of flaked sodium hydroxide, 600 grams of anhydrous sodium pyrophosphate, 600 cubic centimetres of egg yellow colour solution, and 6,000 grams of carub gum. This solution is then mixed with a further 700 litres of skim milk and the whole dried at once on a roller drying machine. The machine is fitted with steel rollers which take about 5 seconds for one complete revolution and which operate under a steam pressure of 7-8 atmospheres. The dried product is then sieved and packed. It has a colour similar to spray-dried whole egg powder but its texture and flavour resemble ordinary roller-dried milk powder.

" Milei W " is an egg albumen substitute prepared by mixing 5,200 litres of skim milk with 1,000 litres of whey and 23·2 kilograms of slaked lime. This is concentrated to a syrupy consistency and dried in a spray-drier in which the temperature of the ingoing air is 160°C.

and the outgoing air 70° C. The resultant powder is creamy-white in colour, of medium texture, and inclined to cake unless stored in airtight containers.

"Alroha Va," a whole egg substitute from Robert Pöhler of Hamburg, and "Synmilka," an egg albumen substitute from Schoppe and Schultz of Hamburg, are two other products manufactured from skim milk. The former, which contains 20-25 per cent. cereal starch, is a creamy powder with a rather coarse texture. The latter, which contains 10-15 per cent. added starch, is paler in colour and of a much finer texture. Manufacturing details are not available for either of these products.

Robert Pöhler has also marketed "Spar-Eipulver," an egg substitute in which only 50 per cent. of the powder consists of a skim milk substitute (probably "Alroha Va"), and "Trocken-Vollei," an unadulterated whole egg powder.

Samples of all these products have been obtained from Germany and used in test bakes on a 5 and 10 per cent. dry matter basis. The results of these bakes are given in Table 13.

TABLE 13.—THE ADDITION OF WHOLE EGG AND EGG ALBUMEN SUBSTITUTES TO BREAD

Reference Number	Substitute	Amount	Bread Score
		%	
Control	.. ..	..	100
104	" Milei V "	5	50
105	" Milei V "	10	60
106	" Milei W "	5	50
107	" Milei W "	10	30
108	" Alroha Va "	5	50
109	" Alroha Va "	10	40
110	" Synmilka "	5	45
111	" Synmilka "	10	40
112	" Spar-Eipulver " ..	5	50
113	" Spar-Eipulver " ..	10	40
114	" Trocken-Vollei " ..	5	80
115	" Trocken-Vollei " ..	10	70

When "Milei V" was added to doughs, the resultant loaves were immature, had a very small volume, and a slightly tough, close texture. "Milei W" also produced loaves which were immature and small in volume but in these loaves the texture was open and there were occasional cores. Both "Alroha Va" and "Synmilka" produced loaves which were immature, poor in volume, had tough textures and contained numerous cores. With "Spar-Eipulver," there was a slight improvement in maturity and volume but both loaves contained frequent cores. The best loaves were those containing "Trocken-Vollei." These loaves were only slightly immature and the volumes good. The loaf containing 5 per cent. "Trocken-Vollei" had a fine, even texture, but the texture of the other loaf was slightly coarse and open.

(xi) Skim Milk Powders Containing Various Percentages of Fat

A number of milk powders were next prepared by homogenizing various amounts of lard into fresh skim milk and spray-drying in a laboratory drier. Before adding the lard, the skim milk, which had an acidity of 0·18 per cent., was heated to 85°C. for 30 minutes. These powders were added to test doughs so that each dough contained 13 grams of powder but a different amount of lard. The results of these tests are given in Table 14. From these results it is seen that the optimum percentage of lard lies within the range of 21-28 per cent. Above 28 per cent. the value of the loaf decreased rapidly, owing principally to a reduction in volume and a slightly open crumb. Below 21 per cent., the value decreased slowly so that a loaf containing 14-21 per cent. lard was a decided improvement on an ordinary water loaf and only slightly inferior to loaves containing 21-28 per cent. lard

TABLE 14.—THE ADDITION TO BREAD OF POWDERS CONTAINING
DIFFERENT PERCENTAGES OF LARD

Reference Number	Lard	Bread Score
	%	
Control		100
116 ..	7	95
117 ..	14	110
118 ..	21	115
119 ..	28	120
120 ..	35	105

Because the addition of fat reduces the percentages of the various skim milk constituents, it is desirable not to add more fat than is absolutely necessary. It was therefore decided that 20 per cent., or slightly less, would be a convenient and effective amount of fat to incorporate in skim milk powders for future tests.

(xii) Skim Milk Powders Containing Lard, Beef Fat, or Mutton Fat

Arrangements were next made with a large milk-drying company for the manufacture of three special batches of skim milk powder. These powders contained approximately 19·6 per cent. mutton fat, beef fat, and lard respectively. Each batch was prepared from fresh skim milk, having an acidity of 0·16 per cent., and into which there had been mixed a percentage of fat equal to one-fourth the percentage of non-fatty milk solids. This mixture was preheated to 85°C. for 30 minutes and homogenized under a pressure of approximately 3,000 pounds per square inch. It was then condensed in a vacuum pan to one-third its original volume and dried in a Merrell-Soule spray-drier, the temperature of the drying chamber being approximately 76°C. and the time during which the powder was held at this temperature about 10 minutes.

These three powders were used in a number of baking tests, the results of these tests being given in Table 15. From these results it will be seen that there is no very marked difference between the baking properties of skin milk powders containing mutton fat, beef

fat, or lard. The powder containing mutton fat produced loaves with the best volume although the volumes of all loaves were good. The crumbs were fine, even, and soft. The powder containing lard produced a crumb with a particularly fine texture and this was largely responsible for the slightly higher scores of loaves containing this powder. All the loaves were of average maturity.

TABLE 15.—THE ADDITION OF VARIOUS AMOUNTS OF THREE SPECIAL POWDERS TO BREAD

Reference Number	Fat in Powder	Amount of Powder	Bread Score
		%	
ontrol	.	.	100
21	Mutton	6	110
22	Mutton	8	115
23	Mutton	10	110
24	Mutton	12	110
25	Beef	6	110
126	Beef	8	110
127	Beef	10	115
128	Beef	12	115
129 .	Lard	6	110
130	Lard	8	115
131	Lard	10	120
132 .	Lard	12	115

In Australia mutton fat is less than half the price of ordinary skim milk powder and much cheaper than beef fat or lard. Hence, in future work, it was decided to use powder containing mutton fat in preference to beef fat or lard.

From the results of the preceding table, it will be observed that there is little difference in the physical properties of breads containing 6, 8, 10, and 12 per cent. powder. In a recent address Dr. F. W. Clements (1946) stated that in order to make a substantial contribution to the nutritional level of the Australian people, it would be necessary to add approximately 10 per cent. skim milk powder to bread. This would be equivalent to approximately 12½ per cent. of a powder containing 20 per cent. fat. It was at this level that it was decided to conduct further work on the development of a standard skim milk loaf.

3. Commercial Tests

Arrangements were made for the manufacture of another much larger quantity of skim milk powder containing slightly less than 20 per cent. mutton fat. The details of manufacture were similar to those already described. This powder was distributed to a number of commercial bakeries for the production of 2-lb. " tin " or " Devon " loaves, about 75 per cent. of the bread baked in Australia being of this type. The details of manufacture were left to the individual manufacturers, all of whom were very competent craftsmen. Some bakers gave the loaves a longer fermentation time, others preferred

to add extra yeast, and one baker added a small amount of cane sugar. For each pound of skim milk powder added, approximately 0·8 lb. of extra water was used.

(i) *Physical Properties of Commercial Loaves*

In general, loaves baked under commercial conditions were equal, and usually superior, to the ordinary water loaf produced in the same bakehouse. Where " high top " as well as " tin " loaves were manufactured, a small but definite increase in volume was noticeable. Also, the loaves containing skim milk powder were more symmetrical in shape.

In most bakeries, the skim milk loaves had a more pronounced colour, the crumb being quite creamy and the crust a dark straw colour. Some bakers thought this detracted from the value of the bread and they suggested that loaves containing such a high percentage of milk powder should be baked at a slightly lower temperature. The loaves were of average maturity and the textures of both the crust and crumb were good.

(ii) *Aroma and Palatability*

All the loaves containing skim milk powder were neutral in flavour and the layman found it difficult to differentiate between them and the ordinary control loaves. Nevertheless, most bakers were of the opinion that a better tasting loaf could be produced if beef fat or lard replaced the mutton fat.

Several bakers commented upon an unclean fatty odour which was given off during the baking process. This odour was scarcely noticeable in the finished loaves although it stresses the importance of utilizing only the highest quality fats in the manufacture of these special skim milk powders.

(iii) *Yield*

In previous experiments conducted at the William Angliss Food Trade School, West and Wilkinson (1946) added 3 per cent. skim milk powder to the dough mix. Neither the powder nor the remainder of the dough contained additional fat. This amount of powder increased the bread yield but the increase was not sufficiently great to offset the extra cost. For such a loaf to be profitable, therefore, it would have to be sold at a higher price. Although the present tests were conducted under totally different conditions, the conclusions arrived at are the same.

During the present investigation, several bakers costed their bakes and from the results of these costings it would appear that, when the milk powder was added on a 12½ per cent. dry matter basis, the extra yield obtained per bag of flour (150 pounds) was in the vicinity of 25 ‘pounds of bread. This extra yield paid for little more than half the additional cost of adding milk powder. Hence, such loaves are economically possible only when they are sold at a slightly higher price.

4. Conclusions

Although the simultaneous addition of emulsified fat appears to overcome the major technical difficulty in the incorporation of skim milk powder in Australian bread, several problems still remain.

Because of an increased fermentation time, loaves containing 10 per cent. skim milk solids take approximately one hour longer to prepare. This can readily be overcome by adding cane sugar or additional yeast although a more satisfactory solution could probably be achieved by the introduction of a suitable lactose-fermenting yeast. During the latter part of this investigation, fat was very finely and evenly distributed throughout the dough mix by homogenizing it into the skim milk before converting it to powder. This was only one way in which satisfactory results could be attained. Commercially, it may be more desirable to add emulsified fat as a separate ingredient. Also, it has been apparent that the introduction of several small modifications in the baking technique would assist in producing a more satisfactory loaf.

In the near future, it is hoped to commence further work in which these problems will be investigated.

5. Acknowledgments

6. References

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An Electrolyte Resistance Thermometer for Measurement of Soil Temperature

*By C. G. Gurr, B.Sc.**

Summary

A resistance thermometer, using sodium chloride solution as the resistance element, has been found to have sufficient sensitivity for soil temperature to be measured with a standard commercial Wheatstone bridge.

Some means of measuring soil temperatures has been found desirable in connection with measurements being made of the water content of soils by an indirect method involving the measurement of the electrical resistance of gypsum blocks embedded in the soil. For convenience in field use it was desired to use the same Wheatstone bridge for both block resistance and temperature measurement. However, the bridge in use for the former did not have sufficient sensitivity for use with the usual metallic resistance thermometers.

Since the temperature coefficient of resistivity of electrolytes is so much greater than that of metals, being of the order of 2 per cent. per degree Centigrade, an electrolyte resistance thermometer was developed which on the same bridge measured temperature to an accuracy of approximately one quarter of a degree Centigrade. The greater accuracy of thermocouples and platinum wire resistance thermometers when used with appropriate measuring instruments is offset by the simplification of apparatus for the electrolyte resistance thermometers, where a high degree of accuracy is not necessary.
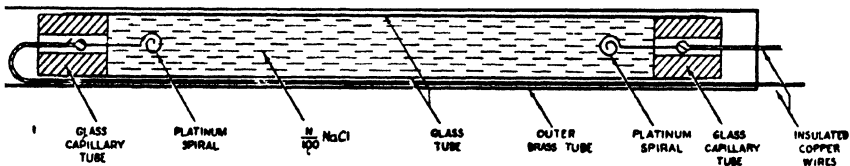
FIG. 1. —Construction of electrolyte resistance thermometer.

The form of the thermometer is shown in Fig. 1. The electrodes consist of platinum wire spirals fused onto copper leads which are cemented into short pieces of capillary tubing. The capillary tubes are in turn cemented into a four-inch length of glass tubing which contains the electrolyte. The whole is placed in a five-inch length of brass tubing for mechanical strength, and the ends sealed with wax.

The solution used was N/100 sodium chloride, which gave a resistance of the order of 20,000 ohms, falling in the most sensitive range of the bridge used. After manufacture the units required aging, since they increased in resistance at a given temperature until reaching a constant value in approximately ten days when the aging was done at 30°C.

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At the low frequency (50 cycles per second) of alternating current used in the bridge, these thermometers have appreciable capacitance, which was balanced out to obtain the most sensitive resistance readings. Plotting resistance on a logarithmic scale against temperature as in Fig. 2 gave a straight line over the temperature range used, so that three points only were necessary for calibration of each thermometer over the range of 5°C. to 30°C.
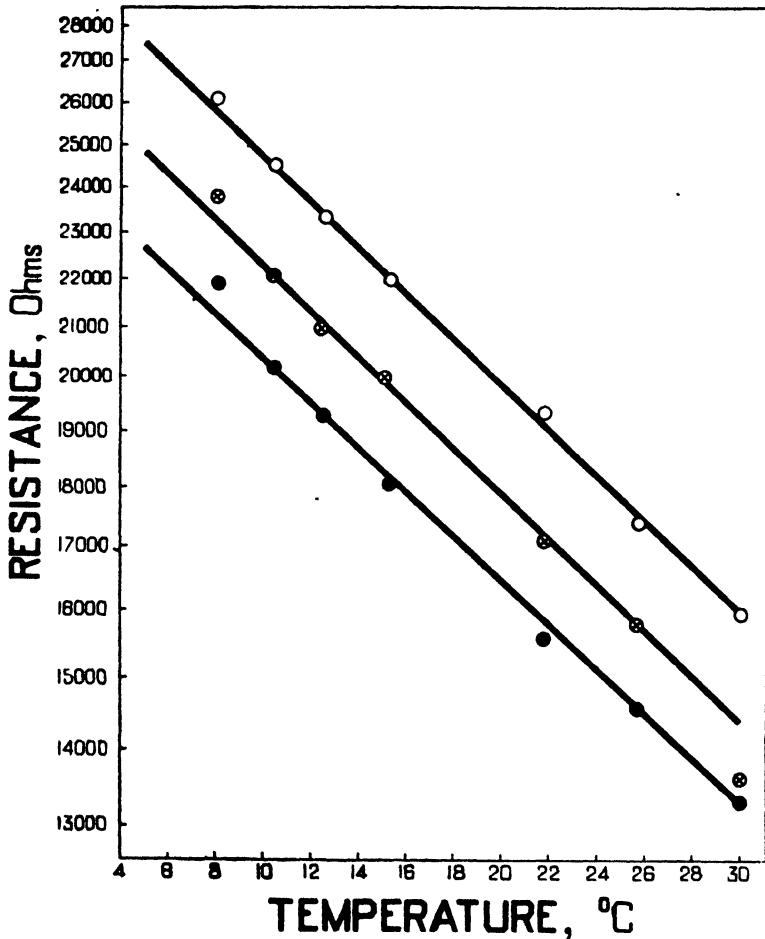
FIG. 2.—Calibration curves of three electrolyte resistance thermometers.

Owing to the high heat capacity of the thermometers there is a lag in response to temperature changes. At the depths of soil at which these units are being used, temperature changes are slow and the lag is not significant. For surface soils where temperature changes may be more rapid, this lag could introduce errors in temperature measurement.

Since these units were constructed, Bouyoucos (1947)* has published an account of a similar type of thermometer, using organic liquids as the resistance element, which can accommodate a much greater temperature range than the aqueous salt solution used above. The Bouyoucos units have other desirable features, such as platinum electrodes fused to the glass envelope, and small size, giving low heat capacity and hence less lag in response to temperature changes. These features should make the organic liquid thermometers more suitable for general use. However, for the soil conditions at present being examined, the thermometers developed in this laboratory have proved satisfactory.

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* Bouyoucos, G. J. (1947).—New electrical resistance thermometer for soils *Soil Sci.* **63**: 291-8.

The Water Absorption of Phenol-formaldehyde Resin Mouldings

By R. P. Bowman,* J. S. Fitzgerald, M.Sc., Ph.D.,† and Florence M. Jensen, A.M.T.C.†

Summary

Published work indicates that the absorption of water by plastics is a diffusion process involving two independent factors—the rate of penetration and the saturation limit. With phenolic mouldings in particular, the process is so slow that specimens of the size used in the standard water absorption tests are far from saturated after the normal immersion period. It is inferred that the standard tests give an approximate comparison of the rates of penetration of water into various plastics, provided they do not differ greatly in their saturation level.

It is shown that water absorption by moulded phenolics conforms to the diffusion equation with an accuracy that depends on the filler used. Thin specimens may be used to obtain a measure of the saturation limit of mouldings, but the total percentage of water absorbed varies with the thickness of the specimens.

In the standard B.S.S. and A.S.T.M. tests the results are sensitive to the moisture content of the moulding powders and to the treatment of moulded specimens before conditioning.

It is concluded that for satisfactory comparison of plastics the rate of penetration of water and the saturation value must both be measured. However, if the present standard type of test is retained, more details should be specified than are given in either B.S.S. 771 or A.S.T.M. D570, particularly with regard to the moisture content of the powder before moulding, and treatment of specimens. The A.S.T.M. procedure is preferred to the B.S.S., but results should be expressed as a rate of penetration.

I. Introduction

In Australia the physical, mechanical, or electrical properties of plastics are determined according to methods developed either by British or American testing authorities. During the war there has been an increasing use of British Standard Specifications, particularly by the Services, while manufacturers have used both British and American methods in controlling the quality of their products.

Now that the establishment of Australian standards for some plastics is under consideration, it is necessary to examine critically the existing methods of test. German procedures should be taken into account as well as British and American, though there has been far less experience of them in Australia. Each test procedure will need separate consideration, for it is only to be expected that differing methods will each contain desirable features. Moreover there is some confusion in the application of standard tests, largely due to differences in their purpose. Test methods of the British Standards Institution have usually been developed for a particular type of material, whereas those of the American Society for Testing Materials are applied to a wide range of plastics. The result is that some British books on plastics describe the British tests for certain properties, but in comparing different materials they list results found, apparently, by the American methods (1, 2).

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, If other than an arbitrary choice of method is to be made, a review of available literature supplemented by a certain amount of experimental work will be needed in connexion with each test examined. The present paper deals with the measurement of water absorption of moulded materials, particularly moulded phenol-formaldehyde resins, examining the standard British and American tests in the light of published work and further experimental data.

2. Previous Work on Water Absorption

(i) *The Factors Involved*

A considerable amount of work has been directed to the study of the penetration of water into solids, and the theoretical aspects of the diffusion process have been thoroughly examined (3). Possibly the best discussion of the subject as it applies to plastics is that given by Irany (4) based largely on his experiments with various polyvinyl plastics. He describes the two types of absorption, diffusion, and capillary penetration. The latter type may be represented by equation (A) which gives a parabolic curve, implying a breakdown of the material before reaching saturation.

$$W = C\sqrt{t} \ \ldots\ldots\ldots (A)$$

where W is the total absorption per unit area at time t and C is a material factor.

For absorption when the rate is governed by diffusion Andrews and Johnston (5) and later, but independently, Irany (loc. cit.) developed similar expressions from Fick's law, which showed that the amount of water entering a solid was dependent on two factors, the rate of penetration and the saturation limit.

When the solid considered is in the form of a sheet, the expressions from both papers may be written as equation (B).

$$\frac{W}{S} = 1 - \frac{8}{\pi^2}\left(e^{-Kt} + \frac{1}{9}e^{-9Kt} + \frac{1}{25}e^{-25Kt} + --\right)\ldots\ldots(B)$$

when $K = \dfrac{k\pi^2}{d^2}$ (Andrews and Johnston) or $= \dfrac{P}{d^2}$ (Irany),

d is the thickness of sheet,

W is the total water absorption per unit area at time t,

S is a constant depending on the saturation limit of the material under the conditions of the test,

K, P, k are constants depending on the permeability of the material.

The absorption of water by most homogeneous resins accords with equation (B), and hence follows the diffusion law. Andrews and Johnston (5) showed this held approximately for rubber, and later Leopold and Johnston (6) found it held accurately for " clear Bakelite." Irany (4) showed that diffusion was the governing principle in absorption by various polyvinyl acetates and acetals, but when these contained a filler the absorption rates followed the capillarity equation (A), even though the filler was a non-absorbent mineral powder. He also showed that the same equation held for a laminated Bakelite which contained a fibrous filler. Though Taylor (7) reported phenolic

resins and vulcanized rubber as reaching equilibrium (saturation), he found that in most cases water absorption followed the capillary law up to a point since the water absorbed increased as the square root of the time. Some of the results given by Kline, Martin, and Crouse (8) were used by Irany as examples of diffusion (see Table 2 later), but for the cast phenolic resin the original figures, with the exception of the final two measurements, fit a single constant parabolic curve as well as they do the curve drawn from Irany's two constants.

A number of workers who have studied water absorption of various plastics, including materials containing fillers, have stressed that two factors are involved, saturation level and permeability. It is therefore of interest to examine the literature in which these factors have been distinguished, and to find what methods have been used to discriminate between them, and what significance has been placed on them. This is particularly necessary since both the British and the American standard test methods lead to but one numerical value as a measure of absorption.

(a) *Rate of penetration.*—Irany used 4 in. by 1 in. specimens of various thicknesses sealed at the edges, which were soaked for prolonged periods, removed and weighed at intervals. To measure saturation, very thin specimens were used, of the order of 0·4 mm. thickness, while specimens for the measurement of the rate of absorption were approximately 2 mm. thick.

In most cases of practical interest the rate of penetration is slow, and most of the water remains near the surface unless the exposure is sufficiently prolonged. Irany therefore concluded that experiments based on such short immersions as that accepted by the A.S.T.M., namely 24 hours at 25°C. for a specimen 0·125 in. thick, yield comparative rate values but no information on absorption capacity. The scope of A.S.T.M. method D570-42 (9) is specifically limited in its first section to "the procedure for determining the relative rate of absorption of water." However, Note 2, later in the' same section, causes confusion when it states that the test is "a guide to the proportion of water absorbed by a material——."

The water absorption test of B.S.S. 771 (10) specifies a thicker disc, approximately 0·4 in., and 7 days' immersion at about 20°C. It is not stated that only the rate of absorption is indicated and not the saturation limit. Test pieces of the same thickness as the B.S.S. specimens were used by Pinten (11), who soaked mouldings 100 mm. by 15 mm. by 10 mm. in size. He recognized that it is not possible to obtain a complete and reliable evaluation of water absorption on the basis of tests for such short periods as 7 days, for this, he said, gives only a velocity characteristic of the material, whereas saturation value is also important. Nevertheless he classified plastics according to their absorption in this period.

Kline, after a more general paper which summarized existing tests (12), published with Martin and Crouse (8) what is probably the most comprehensive work on water absorption. This paper provided the experimental basis for the present A.S.T.M. test. Using a selection of thermosetting and thermoplastic materials, these workers investigated water absorption during short and long immersions as well as the effects of conditioning the moulding in several ways. Their

results do not point to any one set of conditions as outstandingly
suitable for a test. Even with those adopted by A.S.T.M. and incor-
porated in test D570-42, certain limitations must be borne in mind in
interpreting the results.

From prolonged immersion tests Kline showed that for most of the
materials maximum absorption was not reached until about 16 weeks
or longer, though cellulose acetate and nitrate reached a maximum in
under 8 weeks, possibly in one week. It is obvious that immersion for
24 hours, the time adopted for the A.S.T.M. test, measures only the
initial rate of absorption and has no direct relation to saturation value;
indeed the relative order of materials may be entirely altered by
changing from one basis to the other, as is shown by the following
extract from Kline's results.

TABLE 1.—WATER ABSORPTIONS (Kline, 8)

Material	Percentage Gain during Immersion	
	24 Hours	100 Weeks
Phenolic cast	0·24	7·0
Methyl methacrylate	0·33	1·5
Phenolic moulded	0·34	6·8
Urea moulded	0·75	5·2
Ethyl cellulose	0·83	4·0
Cellulose nitrate	0·98	(2·1)*
Polyvinyl butyral	1·07	9·8
Cellulose acetate	2·5	(5·2)*

* Seven days' immersion (maximum value recorded).

Too few data were given by Kline, Martin, and Crouse to permit
the saturation values to be calculated for cellulose nitrate or acetate
since plasticizer loss between one and eight weeks exceeded any gain in
water. The fractional saturation reached in 24 hours by these materials
and by a moulded phenolic shows, as an extreme example, that an
arbitrary choice of time gives no comparable basis for different
plastics. Lawton and Nason (13) gave water absorption figures for
cellulose acetate and cellulose nitrate specimens 0·125 in. thick and
less, which showed saturation values of 8 and 2·5 per cent. by weight
respectively. In the first 24 hours' immersion the two materials took
up 2·08 and 0·98 per cent. water, that is, 25 and 40 per cent. of their
total absorption. Figures in Table 1 show the phenolic moulding
absorbed only 0·34 per cent. water in the first 24 hours, that is, 5 per
cent. of its saturation capacity, which was 6·8 per cent. of its weight.
Comparison of the different materials if made on the results of 24-hr.
immersion, would be influenced more by rates of absorption than by
saturation values, but would give a true picture of neither.

Some of Kline's figures were examined by Irany, who derived the
constants shown in Table 2 for his diffusion equation (B).

TABLE 2.—CONSTANTS FOR DIFFUSION* (Irany, 4)

Material	S (Saturation)	P (Permeability)
	Vol. %	Sq.cm./day
Methyl methacrylate	1·5	0·0023
Phenolic cast	8·6	0·0002
Polyvinyl butyral	10·3	0·0003

* These constants give only poor agreement with the original figures.

These figures emphasize the conclusion implicit in Kline's work, that saturation and permeability place materials in very different relative positions. They indicate, moreover, that the 24-hr. absorption does not correspond to the permeability constant in Irany's equation. Sufficient figures from strictly comparable materials are not available to make detailed comparisons, but Irany's permeability coefficients would place materials in substantially the same order as constants found for the diffusion of water vapour through plastics.

(b) Saturation value.—From the literature it would seem that the existing type of test gives an approximation to the rate of water absorption, which satisfies the practical requirements of a specification limited to similar plastics. Moreover, it would at first appear that the saturation value, reached only after very prolonged immersion, is mainly of theoretical interest. The effects of the two factors, permeability, and saturation value, are described as follows by Irany:— " A high saturation limit, S, indicates a high water concentration in the exposed surface and, eventually, deleterious effects in such applications which depend mostly on the permanence of the surface (for example in protective coatings). On the other hand, a high rate of penetration, P, would not necessarily affect the surface, but would favour the rapid admission of water to the interior of more bulky objects and cause changes in shape, dimensions, strength, dielectric, or other vital properties." Support is therefore given to the present standard type of test for most applications. However, the necessity for a means of measuring saturation value is shown by such papers as those of Mellon (14) and of Gordon, Brown, and McGrory (15), who considered that for protein plastics only the saturation value was of use. Mellon obtained his measurements in a relatively short time by reducing the thickness of the A.S.T.M. specimen to 1/16 inch, and he noted from a few tests that the method might be suitable for cellulose acetate, urea—and melamine—formaldehyde resins, but not for phenolics.

A method which could be used for measuring saturation absorption is indicated by Shearman and co-workers (16). Adopting a new approach to the problem, they attempted to evolve tests for specimens cut from moulded articles, instead of using specially moulded test pieces. Miniature specimens were tested, and for water absorption, filings were also used. For the absorption tests, specimens 20 mm. by

10 mm. by 0·15 mm. were cut as shavings and immersed for 14 days. In general, materials were placed in the same order as by the standard B.S.S. test if results were expressed as absorption per unit area—a finding which seems anomalous, for one would have expected such thin shavings to have reached saturation. However, comparison of absorptions on a weight basis for small specimens immersed in water and for filings exposed to high humidity, gives some interesting results. The figures given in Table 3 have been taken from the report as an illustration, and it is obvious that the effects both of permeability and saturation are involved.

TABLE 3.—WATER ABSORPTIONS (Shearman, 16)

Material *	In Water		In Air at 100 Per cent. R H
	Standard	Small	Filings
	Mg./sq.cm.	Mg./100 mg.	Mg./100 mg.
Phenolic, wood-flour filled ..	3·24	1·74	15·1
Phenolic, shock-resistant ..	6·70	6·26	12·65
Urea, moulded	9·42	9·55	16·2

* A description of the materials has been given in preference to the trade numbers used by Shearman

Though Shearman and his co-workers do not clearly distinguish between rate of penetration of water and saturation value, the two are obviously of differing importance in the various methods used. From their results the authors conclude that the filings method gives values characteristic of the material rather than of the moulding, since the features of the moulding mainly effective in preventing penetration of moisture have been destroyed by the reduction to powder. They state that both the filings method and that involving immersion of specimens will need to be used if full information regarding both moulding and material is required. It would be better, in our opinion, to interpret the results in terms of saturation and permeability. The differences found depend primarily on surface area and not on other characteristics of the moulded article as distinct from those of the material itself.

Measurements of absorption in atmospheres of high relative humidity were compared by Zebrowski (17) with water absorptions obtained by immersion. He showed that phenolic mouldings exposed to a saturated atmosphere for a period of four years, absorbed water more slowly than by immersion, but ultimately attained a similar value.

Thus two methods have been proposed for obtaining the saturation limits óf water absorption: exposure of filings to atmospheres of high relative humidity, and immersion of thin specimens. The work of Shearman throws doubt on the validity of this second method, but we will discuss it later in conjunction with some of our own results.

(ii) *The Standard Tests*

Assuming, now, that the short time immersion test with moderately thick specimens gives the comparative rates of absorption of water with sufficient accuracy for limited comparisons, it has yet to be ascertained whether the standard tests as described in A.S.T.M. and B.S.S. specifications are sufficiently defined. The details of the tests are set out in the experimental section of this paper, and in what follows certain aspects will be discussed in the light of published information. It is realized that there must be much information in the files of testing laboratories in America and England, but so far as we know this has not been collated.

(*a*) *Expression of results.*—We have concluded above that the standard type of test measures a rate of absorption, and it follows that this would be more explicit if the test results were expressed as a rate. This is of importance since test results are often referred to by users of plastics who have little knowledge of the details of the specifications. B.S.S. water absorption results are reported as the weight of water absorbed. This implies an arbitrary standardization of specimen size, but does not sufficiently emphasize the importance of time. For the A.S.T.M. test, absorption is expressed as a percentage of the specimen weight. This is still less informative; it places the emphasis on weight of specimen rather than on area, and emphasizes the importance neither of specimen size nor of time. The adoption of this mode of expressing results is rather surprising, for it seemed that Kline, Martin, and Crouse (8) had accepted the assumption that absorption per unit area placed materials in their correct relative order, though admittedly they expressed most of their results on a weight percentage basis. Certainly with specimens of constant size the errors caused by using a weight percentage will be of a minor character: they arise from differences in the density of various plastics. The use of the method by Kline, Martin, and Crouse cannot be justified even this way, for they used thicknesses ranging from 0·121 to 0·185 in., giving specimens varying by 6 per cent. at most in area, but in weight up to 40 per cent.

(*b*) *Removal of moulded surface.*—With regard to differences in the experimental technique of the A.S.T.M. and B.S.S. methods, the most obvious is the machining of the specimen for the latter test. There is little published information on this, though the intention seems to be to avoid dependence on surface finish and to simulate worst service conditions. Azam and Luce (18) published a few results for tests on fabric-filled phenolic mouldings which undoubtedly show a greater absorption by sanded specimens than by untreated ones. Their conclusion that full cure reduces the difference between the two types of specimens is not fully supported by their results.

A more satisfactory treatment of this subject is published in a report by the British Electrical and Allied Industries Research Association (19). Their results indicate that specimens with the surface removed by grinding have an increased water absorption, particularly if fabric-filled. The tests were based on B.S.S. 771, but went beyond its usual limit by including polystyrene and thermosetting resins other than phenolics, and also by prolonging the soaking period up to 72 days. It was concluded that the value obtained in the usual short-time test is likely to be a closer approach to the final (saturation)

value if the skin is removed from a moulding than if the skin is retained. This is certainly true, but it can only be of quantitative significance if the values obtained in the short time are of the same order as the saturation values. Results given later in the present paper show this is far from being the case for B.S.S. discs, and in the report cited insufficient time was allowed for saturation to be approached.

(c) *Conditioning of powder and specimen.*—Conditioning of test pieces, before immersion in water, is required by both A.S.T.M. and B.S. specifications, and the merits of the conditioning periods specified will be discussed later. In neither specification are limits placed on the treatment of specimens between moulding and conditioning, nor is any attempt made to ensure that moulding powders are in a standard state before moulding. It is generally recognized that the moisture content of the powder influences the water absorption of the moulding; however, few figures are available, probably because knowledge of how to obtain the best article from a powder has been regarded as part of a successful moulder's art. In two reports by Barwell and Pepper (20) are details of the water absorptions for laminated paper impregnated with a phenolic resin, and dried to different degrees before pressing. There was a very marked decrease in water absorption with increased drying before moulding, but the authors pointed out that " The results obtained after even 7 days' immersion in water are not saturation values and may be interpreted only as evidence of the rate of water absorption."

Recent interest in high-frequency pre-heating of moulding powders has focused attention on the treatment before moulding. Dring (21) has shown that under the conditions of B.S.S. 771, but using specimens moulded after high-frequency pre-heating, there is a decrease in water absorption as compared with normal mouldings, from 107 mg. to 80 mg. for a good wood-flour filled phenolic powder, and from 147 mg. to 101 mg. for a xylenol resin with wood-flour filler. It is possible that the effect on water absorption is wholly due to the drying of the powder, though undoubtedly this method of pre-heating allows a thoroughly hot powder to be used in the mould, with consequent improvement in flow and reduction in curing time. With regard to a closely allied subject, it is of interest to note the graph given by Pinten (11), which shows that post-stoving of specimens (24 hours at 150°C.) not only decreases the rate of absorption but also lowers the saturation value.

(d) *Moisture content of powder.*—It is usually assumed that the powder as received is the material to be tested. However, samples are not always kept in airtight containers, and it may be desirable to condition them to a standard low humidity before moulding. If so, the moisture content of the powder before and after conditioning should be determined. No standard procedures have been suggested for determining the moisture content of thermosetting moulding powders. Cornish (22) states that work in the laboratories of Messrs. Halex Ltd. has shown that most methods suggested for determining the moisture content of moulding powders are unreliable. He used the Karl Fischer reagent and obtained satisfactory results with several types of powders. His procedure should serve as a useful guide in the preparation of a standard test.

3. Experimental

The published work just reviewed is insufficient for comparison of B.S.S. and A.S.T.M. methods of determining water absorption, and for deciding whether such factors as moisture content of the moulding powder or storage of specimens after moulding, at present not mentioned, should be delimited. The evidence regarding the usefulness of thin specimens for determining saturation values is conflicting. The tests described in the following sections provide some further information to assist in formulating a standard test, and indicate lines for further work should it be considered desirable to measure the absorption at saturation.

(i) *Materials Used*

The moulding powders used in the preparation of the test pieces were all commercial products of Australian firms except Nos. " E " and " F " which were imported. Some details of their composition are given in Table 4. The approximate impact strengths (B.S.S. Izod) are also given, since water absorption is often taken to a limited extent as varying in the same way as impact strength, though not proportionately. The moisture content of the powder as measured immediately before moulding specimens is also given in this table, though for some tests made at different times other values were found and are quoted with the relevant experiment.

TABLE 4.—POWDERS USED AND THEIR MOISTURE CONTENTS.

Ref No	Type* and Filler	Filler	Resin Type	Izod.	Moisture Content	
					Normal	Dried
		%		ft./lb.	%	%
A	GX Wood-flour ..	42	2 Stage	0·27	4·2	1·8
B	HS Macerated Fabric ..	44	2 Stage	0·9	6·3	3·8
C	MS Mixed Wood and Fabric	46	1 Stage	0·4	5·5	..
D	Mica ..	68	2 Stage	0·12	..	1·2
E	GX Wood-flour ..	..	..	..	5·3	1·95
F	HS Canvas	..	..	(1.0)	5·6	2·4
G	GX Wood-flour ..	42	2 Stage	..	..	..
H	G Wood-flour	42	2 Stage	..	4·6	..
J	Expl. Wood-flour ..	25	1 Stage	0·18	4·0	1·3

* According to B.S.S. 771.

(ii) *The Standard Methods*

German methods for testing moulded plastic materials need not be discussed here for Specification DIN 7701 (23) includes only a qualitative test, namely, immersion for 15 minutes in boiling water, a process which should bring about no significant change.

Quantitative methods are set out in British Standard Specification (B.S.S.) 771-1938 (10) and in D570-42 (9) of the American Society for Testing Materials (A.S.T.M.). Essentially the two tests are similar: they involve moulding a disc, immersing it in water for a given period, drying and determining the increase in weight. Briefly the details which apply to moulded phenolic materials are as follows:

B.S.S. 771-1938, Appendix E.

A disc 50 ± 1 mm. (1·97 ± 0·039 in.) diameter, 12 ± 1 mm. (0·47 ± 0·039 in.) thick, is moulded in a positive type mould under specified conditions of pressure, temperature, and time; cooling in the mould under pressure is allowable, which varies the last two conditions. The skin is removed by machining, leaving a disc 48 ± 0·2 mm. (1·89 ± 0·008 in.) diameter 10 ± 0·2 mm. (0·394 ± 0·008 in.) thick. This is conditioned for 1 hour at 50°C. cooled in a desiccator, weighed, immersed in water at 20°C ± 5° for 7 days, wiped dry, air-dried for 5-15 mins. and weighed. Water absorption is expressed as mg. increase in weight.

A.S.T.M. D570-42.

A disc 2 in. diameter, 0·125 ± 0·007 in. thick, is moulded, suitably in the type of mould detailed in method D647-42T (24), which shows a fully positive mould in which the chase is only 0·020 in. higher than the two forces together. No conditions are given for making the moulding though the 2 in. disc will ultimately be included in D796-44T on moulding practice (25). The disc as moulded is conditioned for 24 hours at 50°C. ± 3° or for 1 hour at 105-110°C. if comparison with other plastics not required. It is cooled in a desiccator, weighed, immersed in water at 25°C. ± 2° for either 2 hours or 24 hours (the latter usually for phenolic mouldings), wiped dry and weighed immediately. The weight increase, expressed as a percentage of the weight of the conditioned specimen, is taken as the water absorption.

It is of interest to note that in the forerunner of this test, D48-39, a 4 in. disc ⅛ in. thick was used. All materials not affected by such temperatures were conditioned for 24 hours at 100°C. ± 5°, and 48 hours' immersion at 25°C. ± 2° was required.

(iii) *Methods Used*

Water absorption determinations were made by the B.S.S. and A.S.T.M. methods. A fully positive mould was used, giving a disc 2 in. diameter. The weight of charge was adjusted to give 0·45 in. thickness for B.S.S. specimens and 0·125 in. thickness for A.S.T.M. To minimize the possible effects of storage before conditioning a strict time schedule was observed. A.S.T.M. discs were placed in the conditioning oven the day after moulding and immersed the next day. B.S.S. discs were machined the day after moulding and conditioned and immersed the next day. Most experiments were done in duplicate but for the long-term soaking and some other tests five specimens were used for each set. The moulding schedule was arranged so that as many treatments as possible were covered each day, and replicates were moulded on different days. Powders were moulded at 3,000-4,000 lb./sq.in. and 160°C. ± 5°, the B.S.S. specimens for 15 mins., the thinner A.S.T.M. ones for 6 mins., except where otherwise stated.

After immersion and before weighing, specimens were wiped rapidly with damp and with dry cloths, left on a three-point suspension under a crystallizing dish to dry in still air for 5 minutes and then weighed. Discs immersed at 50°C. were left 15-45 mins. in water at 25°C. before weighing. Immersion was normally in distilled water at 25°C. ± 1°, specimens being held on edge in slots cut in a bent piece of methyl methacrylate sheet.

Moisture contents of powders were determined by heating 2-4 g. samples at 100°C. for 24 hours, using shallow aluminium dishes covered by lids during the cooling and weighing. Dishes were cooled on a metal block in a desiccator for 10 mins. and weighed immediately.

(iv) Drying Wet Specimens before Weighing

The difference between the details given in the A.S.T.M. and B.S. specifications concerning the drying of the discs after soaking and before weighing probably originated because of the rougher surface of the machined discs in comparison with the smooth mould finish. A.S.T.M. procedure is to remove the surface water with a dry cloth and weigh immediately, whereas B.S.S. specify a thorough drying by pressing with blotting paper, followed by more than 5 but less than 15 minutes' drying in still air at 15-25°C.

Materials that gave a range of water absorptions were tested. Discs were weighed at intervals after wiping with a towel. Machined discs required 5 minutes' air-drying before they appeared superficially dry. Results were slightly erratic as might be expected from the nature of the experiment, but the loss during drying varied with the water absorption. The results set out in Table 5 are grouped according to the range of water absorption, and are sufficiently accurate only to show the order of the error.

TABLE 5.—DRYING OF DISCS BEFORE WEIGHING

Disc	Absorption	Drying Error in Interval		
		1–4 Mins	1-11 Mins.	1-20 Mins.
	mg.	mg.	mg.	mg.
A.S.T.M. ..	2–15	0·1	0·2	0·3
,,	40–140 ·	0·3	1·5	3·0
		5–10 Mins.	5–15 Mins.	5–30 Mins.
		mg.	mg.	mg.
B.S.S	100–120	2·0	3·5	6·0
,,	150–200	4·0	6·0	9·0

(v) Absorption during Prolonged Immersion

A test was made to find the effect of prolonged immersion on the water absorption of different types of phenolic mouldings.

Materials A-D (Table 4) were used. Conditioning for both B.S.S. and A.S.T.M. size of specimens was 24 hours at 50°C. Five specimens were used for each series of measurements. Owing to the numbers involved, up to a week was allowed between moulding and setting up. Immersion was at 25° and 50°C. and specimens were replaced in water after each measurement. With the prolonged immersion of many specimens, particularly at the higher temperature, it was thought preferable to change the water every few weeks. In the later stages the 50° specimens were placed in water in a 1-gallon bottle with a screw lid, and left in an oven. A strong smell of naphthalene developed and the solution became slightly alkaline. No allowance was made for any material extracted by the water.

Most of the results are represented in Figs. 1-3, though some of the points for short-time immersion have been omitted to simplify the figures. In general, the results are of the type expected, with

the absorptions in 24 hours or 7 days only a small proportion of the saturation value, and with the thicker specimens absorbing a greater total weight of water.

The shape of most of the curves points to diffusion being the governing process and this has been confirmed by finding close agreement with a theoretical curve in some cases, and approximate agreement in others. The results did not fit the capillary absorption equation (A) given earlier, and moreover this equation makes no allowance for attainment of a saturation limit. For expressing the results according to equation (B), all its terms after the first may be neglected for values of Kt greater than about $0 \cdot 25$, when W/S is over $0 \cdot 37$. The expression then reduces to the form (C) in which it was used by Irany. It can also be written as shown in (D), the form used by Andrews and Johnston.

$$W = S \left(1 - \frac{8}{\pi^2} \cdot e^{-Kt} \right) \qquad \ldots\ldots\ldots\ldots \text{(C)}$$

$$Kt = -\ln\left(1 \cdot 234 \left(1 - \frac{W}{S} \right) \right) \qquad \ldots\ldots\ldots\ldots \text{(D)}$$

In using this equation we chose a suitable value for S after graphing the results. Observed values of W were neglected when W/S was below $0 \cdot 37$ (since the simplified equation does not then hold), and also when W was nearly equal to S (since small deviations are unduly magnified). Values for the remaining points, expressed as in (D), were then averaged. Division gave K, the permeability constant for the thickness of specimen under consideration, values for which are shown in Table 6. The derived curves are drawn as full lines in Figs. 1-4.
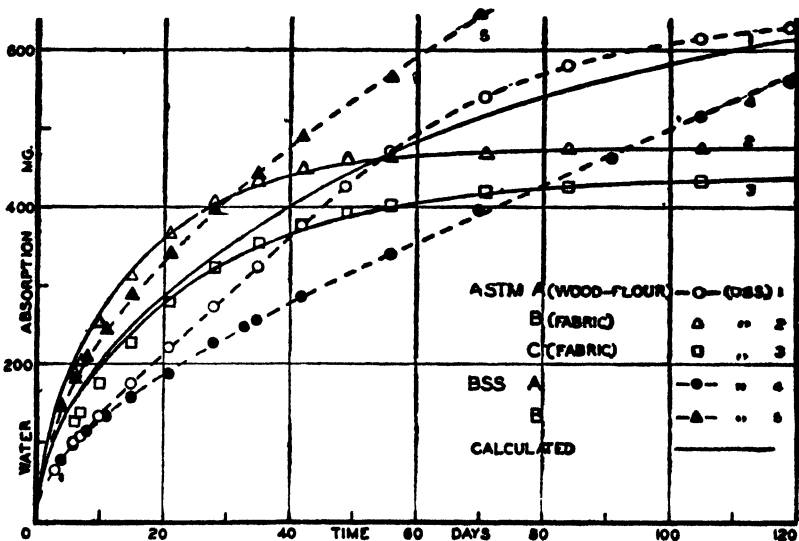
FIG. 1.—Specimens immersed at 25°C.

TABLE 6.—DIFFUSION CONSTANTS (EQUATION B) FOR ABSORPTION.

Powder	Test	Temp	Agreement with Observations	S.	K.	P = Kd²
		°C.		%	1/day	sq. in. /day
A ..	Min. ..	25	Good ..	5·30	0·56	0·00085
	" ..	50	Good ..	5·60	2·11	0·0032
				mg.		
	A.S.T.M. ..	25	Poor ..	700	0·016*	0·00025*
	" ..	50	Poor ..	700	0·10*	0·0016*
A dried ..	" ..	50	Poor ..	925	0·065*	0·0010*
" ..	B.S.S. ..	25	Saturation not approached			
" ..	" ..	50	Poor ..	2,260	0·016*	0·0024*
				%		
B ..	Min. ..	25	Good ..	4·35	0·87	0·0013
	" ..	50	Good ..	4·35	2·50	0·0038
				mg.		
	A.S.T.M. ..	25	Good ..	475	0·060	0·00093
	" ..	50	Good ..	475	0·20	0·0031
B dried ..	" ..	50	Good ..	625	0·12	0·0019
" ..	B.S.S. ..	25	Saturation not approached			
" ..	" ..	50	Fair ..	1,750	0·019	0·0030
C ..	A.S.T.M. ..	25	Fair ..	440	0·039	0·00061
	" ..	50	Good ..	490	0·175	0·0027
D dried ..	" ..	25	Saturation not approached			
" ..	" ..	50	Poor ..	300	0·0088*	0·00014*
J ..	" ..	50	Good ..	800	0·125	0·00195
J dried ..	" ..	50	Good ..	1,100	0·105	0·00165 ·

* Where the curve calculated from these constants agrees only poorly with the points observed, the constants must be taken as approximations only. For comparison of the curves see Figs. 1-4.

Good agreement between experimental results and the requirements of the diffusion equation was not obtained in all instances, and was particularly poor for the wood-flour filled powder. For this material, the graph of results expressed as in equation (D) gave a flat sigmoid curve which approximated to a straight line through the origin. From the graph of absorption against time, it can be seen that, for a considerable portion of the process, absorption by the wood-flour filled mouldings is directly proportional to time.

The deviations from the theoretical shape of curve are small enough to warrant the assumption that diffusion is the main process involved, but that the postulates made in deriving the equation are not all fulfilled. Some capillary action would be expected with the cellulosic filler, but this should be greatest with the fabric filler which nevertheless gave the best agreement with the diffusion equation.

The saturation values were usually similar at 25° and 50°: this was confirmed by using miniature specimens (see Section 3 (vi)). With the wood-flour filled mouldings the higher temperature caused an increased absorption in some instances. The total absorption (saturation absorption) on a weight percentage basis was higher with the larger specimens. For the B.S.S., A.S.T.M. and miniature specimens respectively, 9·4, 8·0, and 5·5 per cent. were found for the

wood-flour and 7·2, 5·4, and 4·4 per cent. for the fabric-filled material. There appears to be no simple explanation for this but it must be kept in mind in formulating a test.
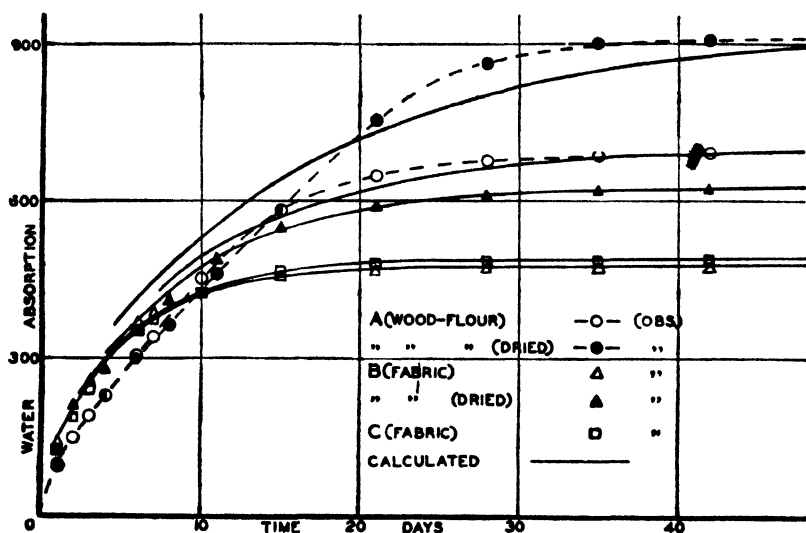
FIG. 2.—A.S.T.M. specimens immersed at 50°C.

In Section 3 (vii) of this paper, figures will be given showing the decrease in water absorption, as measured by the conventional tests, caused by drying the moulding powder before making the test piece. The constants listed in Table 6 indicate that, in the present test, mouldings from pre-dried powders had a lower permeability than those from powders of higher moisture content, but that at saturation they had absorbed a greater amount of water. This difference in saturation absorption may merely reflect the initial difference in moisture content and indicate that at saturation a moulding has a constant water content irrespective of the moisture content of the powder from which it was made. Pre-drying of powders " A," " B," and " J " caused moisture losses of 230, 230, and 240 mg. respectively, while mouldings from the dried and normal powders differed in total absorption by 225, 250, and 300 mg. for the powders mentioned. The significance of this approximate correspondence has not yet been determined.

The reversal of the relative order of different types of moulding materials when compared at saturation and not, as is usual, in the initial stages of absorption, is clearly shown in Figs. 1, 2, and 3 where the curves for materials " A " and " B " cross after 50 days' soaking for A.S.T.M. discs at 25°C., and after a similar period for the B.S.S. size of discs at 50°C.

Cómparison of results obtained with the two sizes of specimen is only valid for the pre-heated powders, all the present B.S.S. specimens having been made with them. Figures for the initial absorptions at 50°C. are given in Table 7.

TABLE 7.—WATER ABSORPTIONS OF B.S.S. AND A.S.T.M. DISCS

Time.	Water Absorption (mg. per disc) at 50° C.			
	Powder " A " (dried).		Powder " B " (dried).	
	A.S.T.M.	B.S.S.	A.S.T.M.	B.S.S.
(days)				
1	97·3	97·4	122·2	151·1
4	228	211	275	303
6	299	270	347	373
8	365	322	408	435
11	460	389	484	511

The greater absorption for the B.S.S. fabric-filled discs (" B ") than for the corresponding A.S.T.M. ones, is probably due to the removal of the resin skin exposing the fibres of the filler and allowing rapid absorption. With the wood-flour filled specimens (" A "), after an initial similar value, the A.S.T.M. disc showed a more rapid absorption despite its smaller area—7·07 sq.in. as compared with the B.S.S. disc's 7·93 sq.in. This may be connected with the difference in curing time. Similar conclusions may be drawn from the constants *P* given in Table 6. Comparisons made after thus allowing for the specimen thickness, show differences which are probably due to factors just mentioned. The agreement between the values for *P* for the high shock resistant moulding, preheated for B.S.S. and not pre-heated for A.S.T.M., is most likely fortuitous.
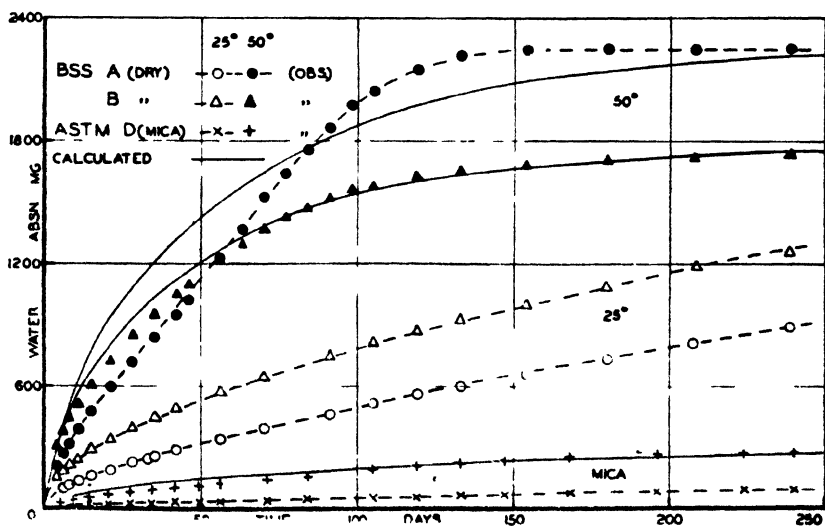
FIG. 3.—Specimens immersed at 25° and 50°C.

(vi) *Tests Using Small Specimens*

Most of our experiments have been made using the conventional mouldings, but a short test was carried out with much smaller specimens. These were prepared by grinding pieces cut from ⅛ in.

thick A.S.T.M. discs in a jig similar to the one described by Shearman (16), and used for making his test pieces for mechanical tests. The size of specimen we used was 40-45 mm. by 10 mm. by 1 mm., both sides being ground.

Powders " A," wood-flour filled, and " B," fabric-filled, were used. The results are shown graphically in Fig. 4.
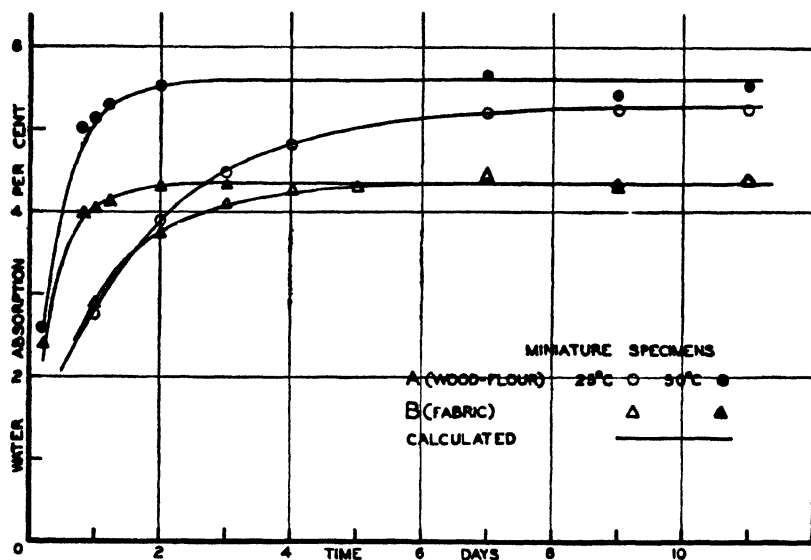
FIG. 4.—Immersion of miniature specimens at 25° and 50°C.

More extensive tests would be necessary before conclusions could be drawn with safety regarding such matters as the differences between total absorptions at different temperatures. The reversal of the relative absorptions of the wood-flour filled and the fabric filled materials between the initial determination and the later ones has been discussed more fully in Section 3 (v), where long immersion tests on full-size specimens are described. It is clear that with the miniature specimen used absorption' is virtually complete in a comparatively short time, only 7 days being required at 25° and 3 days at 50°C. It is unlikely that these periods would be sufficient for water-resistant materials such as mica-filled phenolics.

It seems anomalous that these 1 mm. thick specimens should apparently reach saturation in a week at 25°C. while the pieces only 0·15 mm. thick used by Shearman were apparently not saturated in a fortnight (see Table 3 earlier). It is possible he used surface shavings while his filings were more truly representative of the whole moulding.

(vii) *Effect of Moisture Content of Powder*

As already mentioned, there is no published information on the extent to which the moisture content of a moulding powder influences the water absorption of moulded specimens. If the effect is considerable this factor will be of importance in drawing up a specification for two reasons. First, some conditioning procedure should be laid

down if results of a test are to indicate a property of the powder independent of normal variations of prior treatment. Second, the moisture content of powders may be changed not only by direct pre-heating and such commonly adopted devices as breathing during moulding, but also by chance variation in the time taken to load and close the mould.

To determine the effect of changing the moisture content of a powder, on the water absorption of the resultant moulding, the following experiment was carried out:

The two powders used, "E" and "F," contained wood-flour and canvas fillers respectively. Both powders were used with two moisture contents. Owing to the tendency of the thicker mouldings to become domed soon after pressure was released, mouldings of "E" with the higher moisture content were cooled to 120°C. before ejection, the mould being transferred rapidly to a press with cold platens at the end of the 15 minutes normal cure. With powder "F," all mouldings were ejected hot but immediately pressed between cold platens with 2,000 lb./sq.in. pressure. All A.S.T.M. discs were ejected hot.

Conditioning of specimens was according to the specification—1 hr. at 50°C. for B.S.S., 24 hrs. at 50°C. for A.S.T.M.

TABLE 8.—WATER ABSORPTION AND MOISTURE CONTENT OF POWDER

Material.	Water Absorption (mg. per specimen)			
	"E."		"F."	
Moisture Content (per cent.) ..	5·3	1·95	5·6.	2·4.
B.S.S. (7 days)	166	106	328	167
A.S.T.M. (7 days)	106	102	237	163
„ (1 day)	35	34	70·3	53·2

Table 8 shows that drying powders by pre-heating can cause an appreciable reduction in water absorption. Alternatively this can be regarded as indicating that a powder which initially would have passed the water absorption requirement of a specification, may fail to do so if the initial low moisture content is not maintained.

(viii) *Effect of Conditioning Specimens*

Unless a specification covers the treatment of a specimen from the time it is moulded until the test commences, the conditioning treatment given to a specimen should be sufficient to annul its normal previous history. The B.S.S. conditioning period of 1 hr. at 50°C. is apparently designed to remove surface moisture only, while 24 hrs. at 50°C., as specified by A.S.T.M., is meant to dry the specimen to a standard condition. In neither specification is there any limit to the time which may elapse between moulding and conditioning the specimen.

To find whether the conditioning specified was sufficient to cancel the effect of a severe but by no means improbable treatment of the specimens, the following test was made:

Slightly dried powders were used; "A," a wood-flour filled material, had a moisture content of 1·8 per cent.; "B," a fabric-filled powder, had 4·1 per cent.

The specimens after moulding (and machining for the B.S.S.), were divided into two sets; one was conditioned and tested according to the time schedule adopted for most of our tests, the other set was first stored for 14 days at 25°C. and 75 per cent. relative humidity and then conditioned and tested.

TABLE 9.—WATER ABSORPTION AND CONDITIONING TREATMENTS

	Powder "A."			Powder "B."		
	Absorption.		Decreased Absorption After 14 Days at 75% R.H.	Absorption.		Decreased Absorption After 14 Days at 75% R.H.
—	No Pre-treatment.	14 Days' Storage at 75% R.H.		No Pre-treatment.	14 Days' Storage at 75% R.H.	
	mg.	mg.	%	mg.	mg.	%
B.S.S. (7 days soaking)—						
1 hr. conditioning	96	71	26	197	139	29
24 hrs. conditioning	101	88	13	213	180	15
A.S.T.M. (24 hrs. conditioning)—						
1 day soaking ..	32·2	32·0	0·6	53·2	51·5	3·2
8 days soaking ..	108	94	12·5	183	162	11·5

The results in Table 9 indicate that even when specimens had been conditioned for 24 hours the fortnight at 25°C. and 75 per cent. relative humidity was sufficient to decrease the water absorption over a week by 11-15 per cent. With 1-hr. conditioning the storage decreased the absorption by 26-30 per cent. With the 24-hr. soaking of the A.S.T.M. the difference was only slight, confirming the results of Kline, Martin, and Crouse (8).

Two conclusions can be drawn. If the short period of soaking is accepted the A.S.T.M conditioning period may be sufficient, but even then it would be desirable to place some restriction on the treatment a specimen may receive between moulding and testing. This certainly should be done if the longer soaking period is adopted. The conditioning required by the B.S.S. test is inadequate unless treatment of the moulding is to be rigidly defined.

Choice of the best drying period should be based on a knowledge of the rate of drying of the specimens. Discs of A.S.T.M. dimensions moulded from powders "A" and "B" were dried at 50° or 100°C. and weighed periodically. The results have been graphed in Fig. 5 and show that the loss of moisture is slow and that specimens lose less moisture when made from dried powder. In contrast to water absorption, the wood-flour filled mouldings of "A" lost weight more rapidly during the conditioning period than did those of the fabric-filled "B," and though the powder of "A" contained less moisture, its mouldings lost a greater amount than did those of "B." The constants given in Table 10 were used to calculate the curves shown

as full lines in Fig. 5. These curves agree moderately well with the experimental points, the earlier values being the farthest out, and indicate that diffusion of water to the surface of the disc is the process which governs the rate of moisture loss.
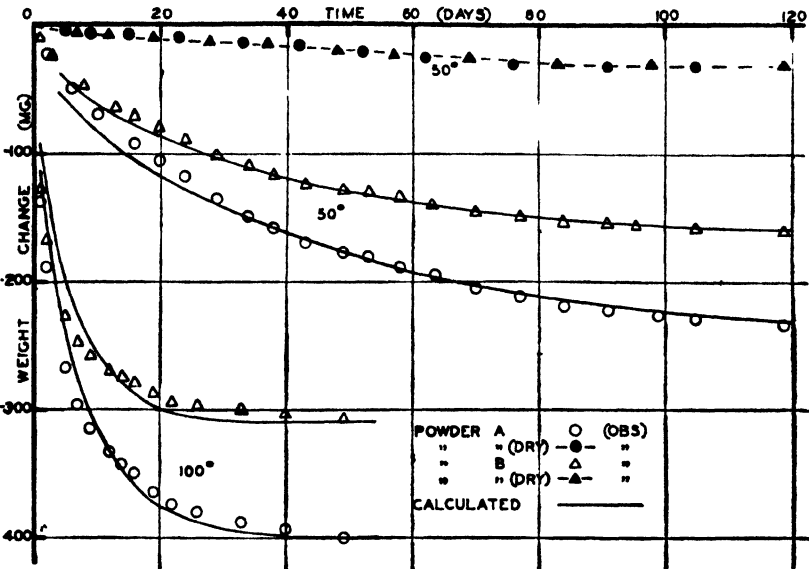
FIG. 5.- Drying A.S.T.M. specimens at 50° and 100°C.

TABLE 10.—DIFFUSION CONSTANTS (EQUATION B) FOR DRYING

Powder	Moisture Content.	Temp.	S	K
	%	°C.	mg.	1/day.
" A "	2·2	50	35	(not calc.)
	5·2	50	245	0·022
	5·6	100	400	0·13
" B "	4·5	50	35	(not calc.)
	7·0	50	165	0·027
	7·8	100	310	0·16

Another aspect of drying is of importance since the A.S.T.M. sets out a procedure for reconditioning specimens after they have been immersed, in order to ascertain whether there has been any loss of water-soluble material. In view of the very slow loss of moisture from specimens made from dried powders, we measured the moisture losses after discs had been immersed in water for 24 hours or for 7 days after the standard conditioning of 24 hours at 50°C. The results obtained with powder " J " have been graphed in Fig. 6, and the figures obtained with this material and powders " A " and " B " in the first few days' drying are set out in Table 11. It is apparent that the difference between the conditioned and reconditioned weights is dependent on the moisture content of the moulding powder and not on a loss of water-soluble material.

TABLE 11.—SOAKING AND DRYING SPECIMENS

Powder.	Moisture Content.	Difference from Conditioned Weight After—		
		Soaking 24 hrs. at 25°C.	Redrying 24 hrs. at 50°C.	Redrying 72 hrs. at 50°C.
	%	mg.	mg.	mg.
"A" ..	4·6	28·8	− 2·7	−15·6
	1·4	28·6	4·4	2·2
"B" ..	8·1	52·6	− 5·3	−25·7
	4·1	49·8	7·6	4·6
"J" ..	4·2	70·6	− 3·1	..
	2·3	78·8	8·8	..

(ix) Effect of Moulding Pressure and Use of Pressure Bar

A test was made to find whether the limits of moulding pressure allowed by B.S.S. 771, 1-2 tons per sq. in., made any appreciable difference to the water absorption of the mouldings. At the same time the effect of a pressure bar was examined since this is sometimes used as a means for ensuring mouldings of correct thickness, and its use is not specifically excluded in the A.S.T.M. specification. With
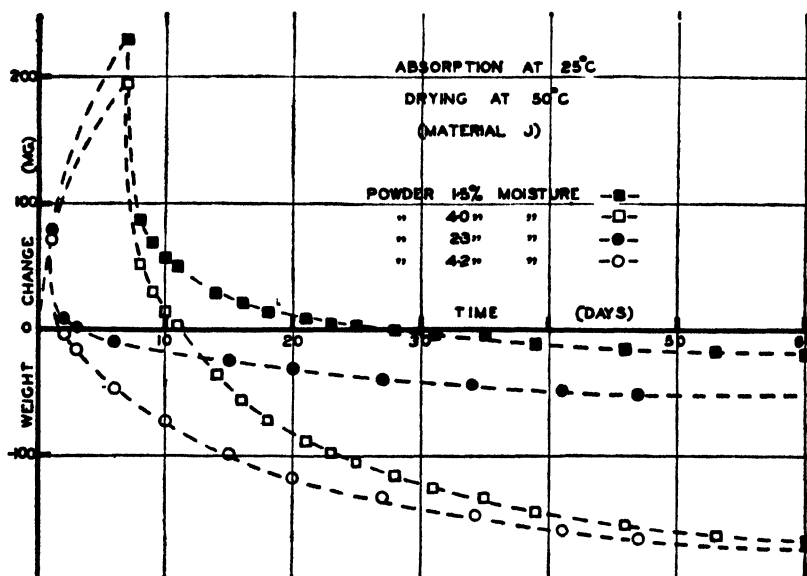
FIG. 6.—Specimens from material " J " immersed at 25° and dried at 50°C.

a pressure bar the platen rests on the bar and chase as well as on the forces when the moulding is of correct size. If the charge moulded is too small, or if material can flow away in the clearance between

forces and chase, the pressure on the moulding may differ from that apparently exerted. In extreme cases an obviously defective moulding will be produced.

In the first test the B.S.S. was followed, but specimens were conditioned 24 hours at 50°C. before immersion. Two samples of a wood-flour filled powder "G" were used, differing in flow properties, but as the mould used gave very little flash from either, the results have been averaged. Two specimens of each powder were used for each condition. Moulding was at 155°C ± 3°, at the pressure indicated, curing being 15 minutes for the hard flowing material and 20 minutes for the soft, the extra time being allowed in order to minimize doming which tended to occur though the moisture contents of the two powders were only 1·6 and 2·1 per cent. respectively, the latter being for the softer material. The charge used at first was sufficient to give a moulding of the desired thickness with no pressure bar in use. The results are given in Table 12.

TABLE 12.—EFFECT OF MOULDING PRESSURE ON WATER ABSORPTION

B.S.S. Type Test with Powder "G"

Charge.	Pressure.	Relative Density.	Thickness.	Water Absorption.	
				1 Day.	7 Days.
g.	lb./sq. in.		in.	mg.	mg.
31·5	2,000	1·337	0·449	50·3	121·6
31·5	4,500	1·338	0·449	46·4	116·8
29·0	,,	1·337	0·412	44·4	113·0
31·5	,, (Bar)	1·338	0·449	47·0	117·9
29·0	,, (Bar)	1·260	0·438	48·4	124·2

The results show a slightly greater absorption with the lower pressure though this gave no significantly lower density. The smaller charge led to increased water absorption and definitely reduced density when a bar was used: the moulding appeared perfect.

A similar test was carried out using A.S.T.M. sized specimens and moulding all for 6 minutes at 155-160° and 3,000 lb. per sq. in. Materials were powders "H" wood-flour filled, and "B" high shock resisting, with moisture contents 4·6 and 6·9 per cent. respectively. The results are shown in Table 13.

TABLE 13.—EFFECT OF WEIGHT OF CHARGE ON WATER ABSORPTION

A.S.T.M. Type Test

Powder.	Charge.	Thickness.		Relative Density.		Water Absorption (7 Days).	
		Bar.	Normal.	Bar.	Normal.	Bar.	Normal.
	g.	in.	in.			mg.	mg.
"H"	9·2	0·129	0·129	1·373	1·374	107·6	107·8
	8·9	0·126	0·124	1·369	1·371	107·5	107·7
	8·6	0·122	0·121	1·356	1·369	114·9	110·4
	8·3	0·122	0·114	1·306	1·373	128·9	113·1
"B"	9·2	0·123	0·121	1·377	1·388	175·3	170·7
	8·9	0·121	0·116	1·369	1·386	175·4	171·4
	8·6	0·121	0·117	1·360	1·390	182·4	167·2
	8·3	0·123	0·109	1·284	1·391	362·3	175·9

The surface finish of the lighter weight discs moulded with the pressure bar was slightly inferior to the others, and one of each duplicate for each material at the lightest charge was incompletely filled out, being crumbly at one spot on the edge. The results show that the use of a pressure bar can lead to decreased densities and increased water absorptions if light weight charges are used, and the mouldings may not appear defective.

(x) *Effect of Temperature of Immersion.*

The temperature of the water in which discs are immersed will obviously have a great influence on the rate at which it is absorbed, though it may not greatly influence the total uptake. Since the standard tests are essentially a measure of the rate so far as moulded phenolics are concerned, temperature control will be of great importance. In the A.S.T.M test the temperature and limits are set out as 25°C. ± 2° whereas 20°C. ± 5° is allowed by the B.S.S.

A simple test was made using powders "G" (wood-flour filled) and "F" (canvas filled) moulded into discs of the A.S.T.M. size. After 24 hours drying at 50°C. these were immersed in water baths at different temperatures, five specimens of each powder being tested at each temperature.

The results set out in Table 14 show that the 10° range permitted by B.S.S. is too liberal and a range of ± 2° is the most that should be allowed for the results to be reasonably accurate, though even this will permit a variation greater than 5 per cent.

The choice of testing temperature must be based on other considerations and in Australia the ambient summer temperatures make 25°C. more simply attained than a lower figure.

TABLE 14.—EFFECT OF TEMPERATURE ON IMMERSION

Powder.	Time of Immersion.	Water Absorption After Immersion at—				
		10°C. ± 1°.	14°C. ± 2°.*	20°C. ± 1°.	25°C. ± 1°.	30°C. ± 1°.
	days.	mg.	mg.	mg.	mg.	mg.
"G"	1	18·1	18·5	24·9	30·0	37·3
	2	25·8	26·8	36·3	43·4	54·4
	3	31·3	33·1	45·2	54·5	69·3
	7	48·3	52·2	72·6	88·8	116·2
"F"	1	31·2	34·7	46·8	57·8	66·3
	2	50·5	54·1	73·6	88·5	103·4
	3	63·5	70·3	95·7	116·0	135·3
	7	102·8	121·6	168·4	204·5	241·3

* Controls on the thermostat were inadequate and average temperature was probably lower than shown.

4. Conclusions

The work at present reported and the papers reviewed lead to conclusions which should assist in the choice of a suitable method for testing the water absorption of phenolic mouldings.

Before setting up a specification test, it is essential that the testing authorities should recognize the two independent factors involved, rate of penetration and saturation level, and decide which is to be measured, particularly if different plastics are to be compared. The present type of test as set out by both A.S.T.M. and B.S.S., indicates only the initial rate of absorption, and results are not comparable over a wide range of plastics. Thin specimens or filings may be suitable for finding saturation values in reasonably short periods, but insufficient work has been carried out to indicate the scope or limitations of such tests. Absorption from a saturated atmosphere is slower than from water, but by either method almost the same saturation level is ultimately reached.

With regard to the conventional method of test, both A.S.T.M. and B.S.S. procedures are sensitive to the history of the moulding powder and moulding prior to conditioning, though the A.S.T.M conditioning does more to eliminate variations than does the short period required by B.S.S. Limits should be set to the time allowable between moulding the specimen and conditioning it for test, and to the humidity to which specimens may be exposed in that period. Moulding pressure should be limited to a narrower range than that allowed by B.S.S. A fully positive mould should be specified.

A disc of the dimensions required by A.S.T.M. is preferable to the thicker, machined disc, for the latter is more difficult to mould with some powders, and the machining achieves no real advantage.

Since an arbitrary time must be chosen for a standard test, 24 hours should be adopted when different types of plastics are to be compared. A longer period, 7 days, is preferable when comparing similar plastics with slow rates of penetration.

Phenolic plastic discs after soaking are not subject to large evaporation losses within the specified period of up to 15 minutes after removing superficial water, though several minutes must be allowed initially for air drying of machined specimens.

For Australian conditions a higher immersion temperature than 20°C. is advisable. The 10° range permitted by B.S.S. is too wide. The A.S.T.M. conditions 25°C. $\pm$ 2° for immersion would be satisfactory.

Results should be expressed as weight absorbed per specimen provided no significant variation of area is allowed. Expressing initial water absorption as a percentage of the weight of the specimen has nothing to recommend it.

An extension of the specification tests to cover a method for determining the moisture content of moulding powders, could well be considered. It should be related to a procedure for conditioning powders to equilibrium with a standard low relative humidity before moulding.

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A Simple Method of Testing Glue Lines in Tension

*By A. W. Rudkin, B.Sc.**

Summary

A method is described for testing glue lines in tension, using, with only slight modifications, the same apparatus as is used for testing glue lines in shear, which is standard equipment in all laboratories concerned with the testing of glued joints.

The test specimens are made up in the form of right-angled crosses in a press-box slightly modified from that normally used for making standard lap joints for shear tests. The modified press-box can still be used for making standard shear strength test specimens.

After reconditioning, the specimens are fitted into a specially designed shackle, which can be fitted into a standard testing machine of the type normally used for shear strength tests. When load is applied to the shackle in the usual manner, it is transmitted to the specimen in a direction normal to the plane of the glue line, so that the tensile failing load can be read off in the same way as shear failing loads are ordinarily read.

Results of tests by this method on a number of types of joints were statistically compared with results of standard shear strength tests on joints made with the same materials, and it was found that the results of the tests in tension were reproducible with at least as high a degree of accuracy as those of standard tests in shear.

1. Introduction

It is well known that practically all types of glued joints are very much stronger in shear than in tension, and for this reason engineers, when designing structures embodying glued joints or laminated members, try to ensure that the glue line is not subjected to severe tensile stresses. Hence most adhesive specifications do not call for tests on the tensile strength of joints, and equipment for this purpose is only available in a few large laboratories.

In the course of a project undertaken by the Council's Division of Aeronautics (Dale, 1945), however, it was found desirable to use laminated members in situations where considerable tensile stresses perpendicular to the plane of the glue line could not be avoided, and hence, when the Division of Forests Products was asked to carry out tests to determine the most suitable adhesives for use in this project, it was necessary to devise means for comparing the tensile strength of joints made with the various adhesives proposed. No apparatus for this purpose was readily available, and some of the materials used in the experiment (Rudkin, 1946), viz. asbestos cement sheet and "Masonite", were not suitable for making the type of joints used for tensile strength tests at, for instance, the Canadian Forests Products Laboratory (Wakefield, 1943).

2. Method

The materials to be glued (mountain ash, asbestos cement sheet, and "Masonite") were cut into slips 6½ in. by 1 in., the length being parallel to the grain in the case of the wood. In one series of tests the slips were 3/16 inch thick; in another series, the mountain ash

slips were ⅜ inch thick, and the other materials, which were supplied in sheets 3/16 inch thick, were glued on to sawn sheets of mountain ash 3/16 in. thick, making up a total thickness of ⅜ inch, before cutting to the requisite size.

The adhesive was applied to the gluing surfaces in the usual way and the slips were placed in the press-box as shown in Fig. 1A, and pressed in the usual manner. This produced a cross-shaped specimen as shown in Fig. 1B, with the glue-line area exactly 1 inch square. A press-box of the type normally used for making standard shear strength test specimens (Standards Association of Australia, 1941) is shown in Fig. 2 for comparison. Fig. 3 is another view of the press-box shown in Fig 1A, containing standard lap-joints for shear tests, demonstrating that the box can still be used in the normal way after being modified to take glue-line tensile strength test specimens.
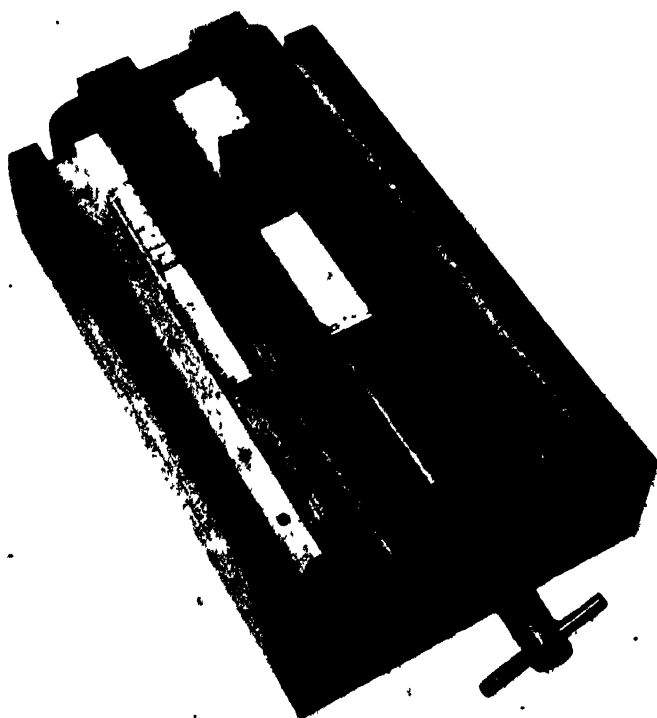
FIG. 1A.—Tensile strength test specimens undergoing pressure in adapted press-box.

After reconditioning, the specimens were fitted into the shackle as shown in Fig. 4, and packed tightly with metal shims to minimize bending stress. The shackle was then fitted into the grips of the testing machine as shown in the figure, and the load was applied in the usual manner until the joint failed. Fig. 5 shows a standard lap

joint fitted into the same machine in readiness for testing in shear, demonstrating that the method of applying the load is essentially the same whether the joint is tested in shear or in tension.
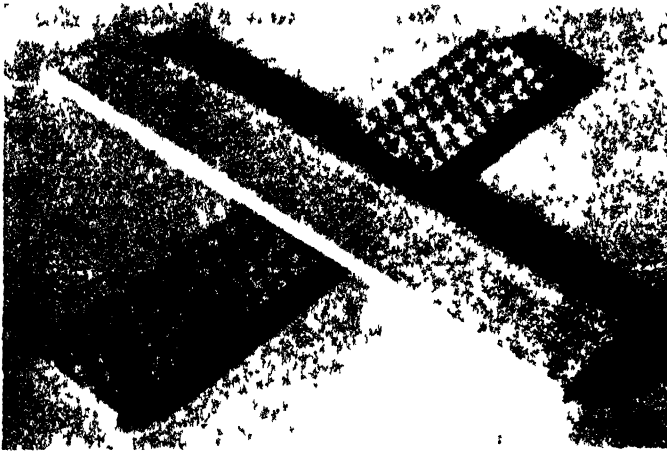
FIG. 1B.—Specimen for tensile strength test.
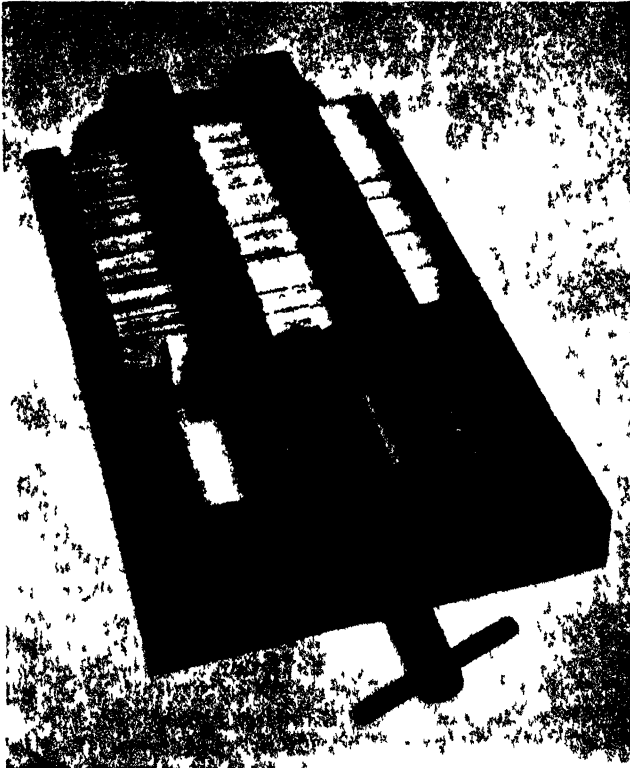
FIG. 2.—Shear strength test specimens in standard press-box.

3. Discussion of Results

An estimate of how accurately results obtained by the method described above correspond to the true tensile strength of the joint could only be made by comparing them with the results obtained with similar materials by methods of proven reliability. This has unfortunately not been possible.
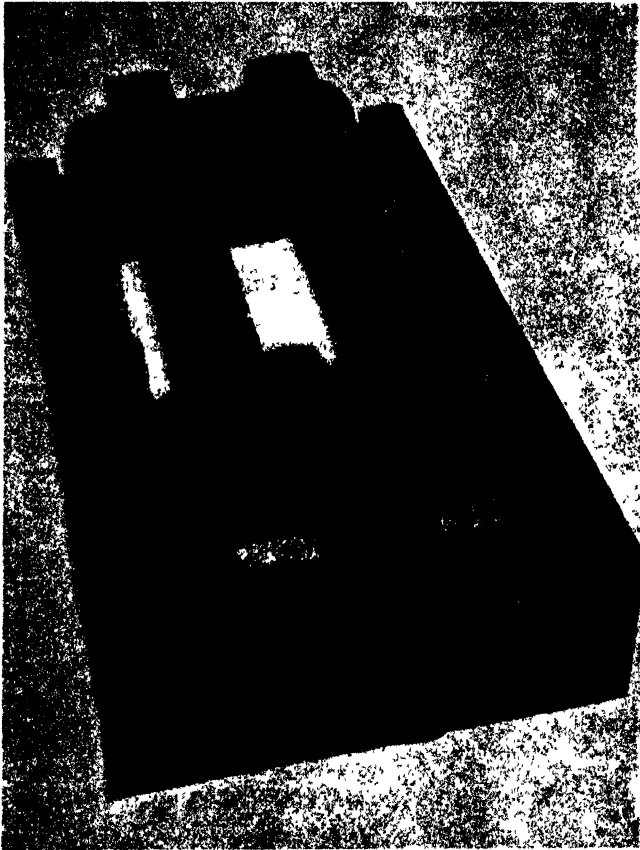
FIG. 3.—Shear strength test specimens undergoing pressure in press-box adapted for making tensile strength test specimens.

Tension tests on the glue lines of phenol-formaldehyde bonded plywood carried out at the Canadian Forest Products Laboratories (Wakefield, 1943) usually gave results of the order of 500-800 lb. per sq. in. on yellow birch, 150-300 lb. per sq. in. on sitka spruce and 300-750 lb. per sq. in. on western white pine; strengths of over 1,200 lb. per sq. in. were recorded on individual specimens. In the tests by the method described in this paper, results on mountain ash (*Eucalyptus regnans*) bonded to mountain ash with good quality wood-wörking adhesives ranged from 138 to 265 lb. per sq. in., and in preliminary tests on coachwood (*Ceratopetalum apetalum*), radiata pine (*Pinus radiata*), rimu (*Dacrydium cupressinum*), and myrtle beech (*Nothofagus cunninghamii*), to check the practicability of the

method, results were usually of the order of 100 lb. per sq. in. Percentage adhering wood fibre was usually slightly higher than in the Canadian tests. These figures indicate that results by the new method may be lower than the true tensile strength of the joints. This may be due to failure to eliminate bending stress to the same extent as in the Canadian tests.

FIG. 4.—Tensile strength test specimen fitted into shackle in testing machine.

Fortunately it is rarely necessary, either in specification testing of adhesives or in more fundamental work on adhesion, to determine the absolute shear or tensile strength of a joint. In specification testing, it is usually sufficient to determine whether a given adhesive gives joints equal or superior to those given by adhesives found suitable

in service tests, and, in more fundamental studies, whether a given change in adhesive formulation or gluing conditions produces a reduction or an increase in joint strength. For these purposes the main desiderata are:—

 (i) The results should be sufficiently accurately reproducible to enable the mean of a small number of tests (usually five in specification testing) to be accepted as a reasonably accurate estimate of the mean value that would be obtained in an indefinitely large number of tests.

 (ii) The results of the test should be positively correlated to a high degree with the true shear or tensile strength as the case may be.

In making standard shear strength tests on plywood or on glued joints, for instance, it has to be remembered that there is no constant value S that can be defined as the shear strength of the glue line in terms of breaking stress per unit area. The failing load, F, of the specimen depends not only on the adhesive, the adherends, the gluing conditions, and the area A, but also on the shape of A and the thickness, T, of the specimen (De Bruyne, 1944). Thus for a rectangular specimen, the value of F is a linear function of the width, W, of the specimen, but not of the length of overlap, L (length being defined as the dimension parallel to the direction of loading), dF/dL tending to very low positive values or to zero as L increases. For fairly thin specimens at least, F also increases with T; again dF/dT tends to very low positive values or to zero as T increases. For these reasons the " shear strengths " of different adhesives, &c., are arbitrarily compared by determining the failing loads of lap joints with a specified overlap (usually one inch square) and made from rectangular slips of a specified length and thickness. The dependence on length of overlap is due to stress concentrations at the ends of the joint owing to differences in *ductility* between the adhesive and the adherends (De Bruyne, 1944); the dependence on thickness of the specimen is due mainly to the fact that bending stresses are not completely eliminated in standard methods of testing.

Hence a proposed method of determining tensile strength can be considered sufficiently accurate for most practical purposes if it can be shown that:—

 (i) Variation among specimens made with the same materials under the same conditions is no greater than that of standard shear strength test results on the same type of joint, thus meeting the criterion of reproducibility.

 (ii) Variation between groups of specimens made with different materials or under different conditions is large compared with variation within the groups, indicating that between-group variation is not random and is therefore correlated with differences between the strength characteristics of the various types of joints.

Results of a series of tensile strength tests are set out in Table 1, and results of a series of shear strength tests on joints made with similar materials are set out for comparison in Table 2. A statistical analysis was made of these to determine whether the method met the above criteria.

The statistical analysis showed that, with specimens ⅜ inch thick, the coefficients of variation of shear and tensile strength test results were approximately equal. The standard deviation was actually smaller in the case of the tensile than of the shear strength test results, i.e. the absolute variability of the former was less than that of the latter (slightly less than one half), even though the percentage variation was slightly greater owing to the much smaller mean values obtained in the tensile tests. The difference in absolute variability was highly significant.
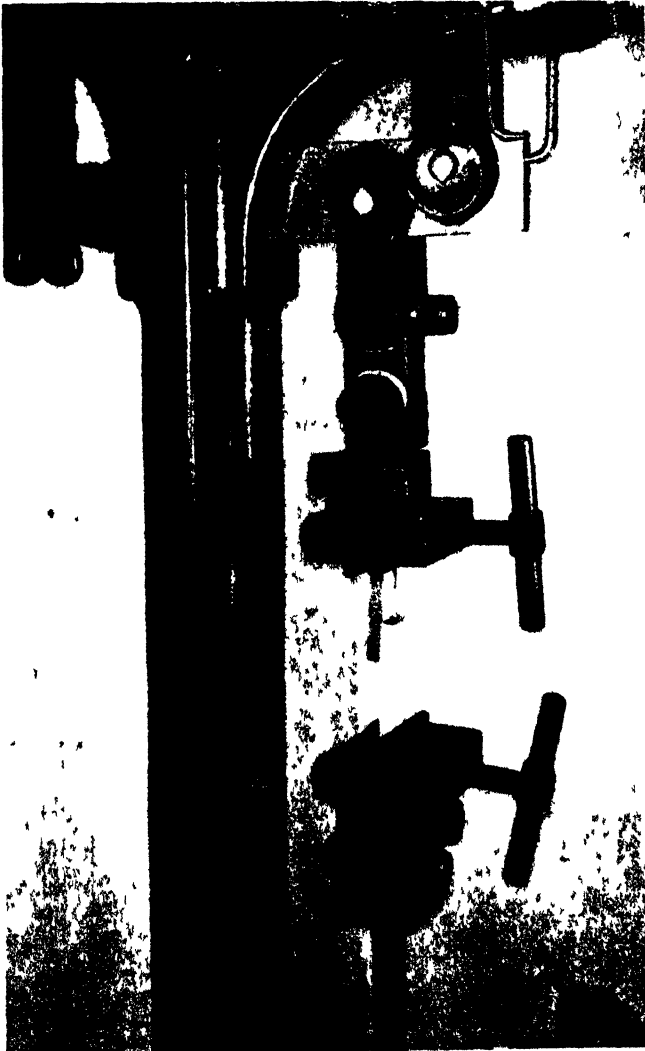
Fig. 5.—Shear strength test specimen fitted into testing machine.

With the specimens 3/16 inch thick, the coefficient of variability of the tensile strength test results was appreciably greater than that of the shear strength test results, the difference being significant at the 5 per cent. level, but even in this case the absolute variability of the tensile test results was rather less than ⅔ of that of the shear test'

Key to Tables

The following abbreviations are used to describe gluing surfaces:—

W = kiln-dried and reconditioned mountain ash.
S = smooth face of asbestos cement sheet.
R = screen (rough) face of asbestos cement sheet.
M = screen face of " Masonite ".

TABLE 1.—TENSILE FAILING LOADS OF GLUED JOINTS

Adhesive	Gluing Surfaces	Thickness of Slips	Number of Specimens	Tensile Failing Loads		
				Min.	Max.	Mean
		in.		lb.	lb.	lb.
Casein cement	W-W	3/16	6	138	265	188·7
„ „		⅛	5	135	254	227·0
„ „	R-W	3/16	3	98	110	103·3
„ „	„	⅛	4	29	103	82·5
„ „	R-R	3/16	3	40	56	46·0
„ „		⅛	5	124	167	141·8
„ „	R-S	3/16	3	18	33	27·3
„ „	„	⅛	5	72	106	96·2
„ „	S-M	3/16	5	43	61	52·6
„ „	„	⅛	5	65	118	97·4
„ „	Coachwood	3/16	5	113	162	134·6
„ „	Radiata pine— Sample A	„	3	94	174	145·0
„ „	Sample B	„	6	61	69	63·0
„ „	Rimu	„	6	107	123	115·2
„ „	Myrtle beech	„	6	80	106	97·8
Rubber cement A	W-W	⅛	5	48	88	72·2
„ „	R-W	„	5	115	144	122·2
„ „	R-R	„	5	78	150	113·2
„ „	R-S	„	5	105	160	122·6
„ „	S-M	„	5	58	126	93·0
„ „	W-W*	„	5	54	132	99·2
„ „	R-W*	„	5	115	159	135·6
„ „	R-R*	„	5	109	150	124·2
„ „	R-S*	„	5	90	137	109·6
„ „	S-M*	„	5	38	131	91·0
Rubber cement B.	W-W	3/16	7	49	83	63·2
„ „	„	⅛	5	55	122	81·4
„ „	R-W	3/16	6	57	78	68·3
„ „	„	⅛	5	92	151	126·8
„ „	R-R	3/16	5	47	68	53·8
„ „	„	⅛	5	53	135	101·2
„ „	R-S	3/16	4	96	131	118·5
„ „	„	⅛	5	97	125	107·6
„ „	S-M	3/16	5	67	90	81·8
„ „	„	⅛	5	62	96	79·6
Liq. phenolic resin	W-W	3/16	5	52	101	77·8
„ „	R-W	„	5	54	90	67·8
„ „	R-R	„	5	45	67	51·2
„ „	R-S	„	4	18	42	28·3
„ „	S-M	„	5	47	83	65·4

* An extended sample of rubber cement A was used.

results, the high percentage variability of the former being due to their low mean. The difference in absolute variability was again highly significant.

TABLE 2.—SHEAR FAILING LOADS OF GLUED JOINTS

Panel Number*	Adhesive	Gluing Surfaces	Thickness of Slips	Number of Specimens	Shear Failing Loads		
					Min.	Max.	Mean
			in.		lb.	lb.	lb.
	Casein cement ..	W-W	3/16	5	660	932	832·2
	" " ..	"	1/8	4	880	1,176	1,074·75
	" " ..	R-W	3/16	6	180	230	210·0
C1FW ..	" " ..	"	1/8	5	128	225	179·8
C2FW ..	" " ..	"	"	5	61	204	151·0
	" " ..	"	"	5	301	460	389·6
	" " ..	R-R	3/16	5	120	143	132·2
C1RR ..	" " ..	"	"	3	73	111	93·3
C2RR ..	" " ..	"	"	4	85	133	100·25
	" " ..	"	"	5	233	306	272·1
	" " ..	R-S	3/16	7	105	215	168·9
C1RS ..	" " ..	"	"	5	45	90	69·0
C3RS ..	" " ..	"	"	5	104	145	130·8
	" " ..	"	"	5	206	302	258·6
	" " ..	S-M	3/16	7	90	228	166·0
C1FM ..	" " ..	"	"	5	103	130	115·0
C2FM ..	" " ..	"	"	5	104	140	126·8
	Rubber cement A.	W-W	1/8	5	116	174	136·8
	" "	R-W	"	5	142	198	180·2
	" "	R-R	"	5	165	186	175·4
	" "	R-S	"	5	193	228	204·0
	" "	S-M	"	5	166	205	188·2
	" "	W-W†	"	5	102	120	112·0
	" "	R-W†	"	5	130	200	167·0
	" "	R-R†	"	5	172	225	200·4
	" "	R-S†	"	5	207	284	239·6
	" "	S-M†	"	5	207	320	279·8
	Rubber cement B.	W-W		4	124	260	192·75
R1FW ..	" "	R-W	3/16	5	62	92	77·8
R2FW ..	" "	"	"	5	64	72	68·2
	" "	"	1/8	5	82	210	134·4
R1RR ..	" "	R-R	3/16	5	117	138	131·6
R2RR ..	" "	"	"	•2	84	89	86·5
	" "	"	1/8	4	136	172	149·75
R1RS ..	" "	R-S	3/16	4	25	76	40·0
R2RS ..	" "	"	"	2	53	125	89·0
R3RS ..	" "	"	"	5	120	133	125·8
	" "	"	1/8	5	137	198	173·0
R1FM ..	" "	S-M	3/16	5	82	120	101·8
R2FM ..	" "	"	"	5	122	152	134·0
	" "	"	1/8	5	137	352	219·0
	Liq. phenolic resin	W-W	3/16	5	640	838	755·8
PB1FW	" "	R-W	"	4	97	170	142·25
PB2FW	" "	"	"	3	163	168	165·3
	" "	R-R	"	3	10	100	61·7
PA1RR	" "	"	"	5	77	128	101·4
PA2RR	" "	"	"	5	90	123	102·0
	" "	R-S	"	3	85	105	93·3
PA1RS	" "	"	"	5	138	197	161·2
PA2RS	" "	"	"	5	152	172	162·8
	" "	S-M	"	6	100	150	126·7
PA1FM	" "	"	"	3	124	193	147·7
PA2FM	" "	"	"	3	104	160	139·0

* Where no panel number is given, the specimens were made up singly and not cut from panels.
† An extended sample of rubber cement A was used.

The analysis further showed that the results on the two thicknesses were consistent with each other, and, on analysing the combined results of the tests on both thicknesses, it was found (a) that there was no significant difference between the coefficients of variation, and (b) that the absolute variability of the tensile strength test results was lower than that of the shear strength test results, the difference being highly significant. The relevant statistical data are summarized in Tables 3 and 4.

TABLE 3.—VARIANCES OF TENSILE AND SHEAR TEST RESULTS

Type of Test	Thickness of Slips		
	⅜ in	1/16 in	Combined
Tensile (lb.)	579 (79)	443 (34)	538 (113)
Shear (lb.) ..	2328 (78)	2917 (37)	2518 (115)
t (Pitman's test) . .	6·67**	6·48**	9·08**

Figures in brackets denote number of degrees of freedom.
** Significant at the 1 per cent. level

TABLE 4.—COEFFICIENTS OF VARIATION AND STANDARD ERRORS OF TENSILE AND SHEAR TEST RESULTS

Type of Test	Thickness of Slips		
	⅜ in	1/16 in	Combined
Tensile (lb.)	21·63 ± 1·60	27·67 ± 3·17	22·86 ± 1·43
Shear (lb.)	19·45 ± 1·43	19·13 ± 2·04	19·34 ± 1·17
Difference	2·18 ± 2·15	8·54 ± 3·77*	3·74 ± 1·86

Weighted mean of coefficients of variation = 20·76 ± 0·91.
* Significant at the 5 per cent. level

The over-all conclusion was that the coefficients of variation of the two types of test (shear and tensile) are approximately equal, the higher apparent coefficient of the tensile strength tests on specimens 3/16 inch thick being a chance result; whilst the absolute variability of the tensile strength test is considerably less than that of the shear strength test. For most practical purposes the absolute variability is more important than the coefficient of variation, most tests of significance being based on absolute and not on percentage deviations from the mean. Hence the proposed test meets the criterion of reproducibility.

The analysis showed also that with the thicker specimens the effects of adhesives and of gluing surfaces, and the interaction between these variables, were highly significant for both tensile and shear tests, and there was a highly significant variation of both tensile and shear strength with differences in treatment. (No attempt was made

to separate the effect of gluing surfaces and of adhesives with the 3/16 inch specimens.) This means it is highly improbable that variations among the results of tests on the different types of joint were due to chance factors, and it is therefore reasonable to assume that they were correlated with the factors being tested, i.e. with the shear strength of the joints in Table 1 and with the tensile strength in Table 2.

4. Conclusion

It may therefore be concluded that the method of testing the tensile strength of glued joints described herein is sufficiently accurate and reproducible for most purposes, i.e., it is probably at least as accurate and reproducible as accepted standard tests on the shear strength of glued joints.

5. Acknowledgments

I am indebted to Mr. J. J. Mack of the Division of Forest Products for his helpful suggestions on the design of the shackle used in the above tests, and for making the working drawing of the design finally agreed upon and supervising the manufacture of the shackle; and to Mr. R. T. Leslie of the staff of the Section of Mathematical Statistics for performing much of the mathematical work involved in interpreting the results.

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The Variation of Maximum Crushing Strength, Maximum Tensile Strength, and Modulus of Elasticity of Hoop Pine (*Araucaria cunninghamii* Ait.) Plywood with Moisture Content

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Summary

Hoop pine plywood in 1/8, 3/16, and 1/4 inch thicknesses, bonded with phenol-formaldehyde, urea-formaldehyde, and casein glues, in each case has been tested in compression and tension parallel, perpendicular, and at 45° to the grain of the outer veneers. The elastic modulus has also been determined from the tensile tests.

The tests were carried out at nominal moisture contents of 8, 12, 16, and 20 per cent. and regression factors determined so that moisture corrections could be made to the corresponding strength figures for this species where necessary.

On account of the inconvenience of application of such factors, and since an additive correction would not be satisfactory in all cases, tables are included giving the strengths at each integral percentage of moisture content over the range of the observations. These are expressed as a percentage of the corresponding value at 12 per cent. moisture content.

1. Introduction

The mechanical properties of wood are influenced by its moisture content, and the law of variation for some static properties at normal temperatures is known to be well represented, for moisture contents below the intersection point, (2, 14) by an exponential relationship (7). For impact properties, the relationship is of a rather different nature and appears generally to follow a somewhat U-shaped curve, often having a pronounced minimum somewhere about 12 to 16 per cent. moisture content (4) although occasionally the curve rises or falls monotonically with increasing moisture content. In the case of tests on solid timber, a considerable variation of moisture content is found to occur from specimen to specimen, even after conditioning at constant temperature and the appropriate relative humidity. It is therefore considered necessary to determine suitable moisture correction figures for species under test and to correct the results of individual tests to a standard moisture content.

Hitherto, no correction for moisture content has been made to plywood test results, since it was generally understood from results of tests overseas that the correction was negligible. This belief is reflected in the Australian standard specifications for aircraft plywood (8, 9), which specify strength values without reference to moisture content except in the " moisture content " paragraph which states that it shall be between 7 and 14 per cent. It has been felt necessary, however, to check this assumption and, should the variation be of such a magnitude as to render correction necessary, to provide correction figures for plywood.

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It was decided that the species to be tested initially for this purpose should be hoop pine. If the corrections should prove of considerable magnitude it might then be necessary to test other species. On the other hand, it is possible that the corrections for solid timber could be applied directly. In this case it is desirable that this conclusion should be checked for one or two additional species.

2. Selection of Material

Twenty logs of hoop pine (*Araucaria cunninghamii* Ait.) were selected from ten trees cut in a number of localities in the Brisbane, Mary, and Gladstone River valleys, the Wide Bay district south of Maryborough, and the Port Clinton district north of Rockhampton. Veneer was peeled from each log in three thicknesses namely 1/24-in., 1/16-in., 1/12-in. The 1/24-in. veneer was peeled at 4–5 in. from the centre of the log, the 1/16-in. at 5–6 in. from the centre, and the 1/12-in. veneer at 6–7 in. from the centre. In this way the effect of distance from pith on the strength properties was minimized, because pith eccentricity was small in all cases. This was considered satisfactory since, in tests on solid hoop pine as an aircraft timber, no significant regression on distance from pith had been found for compressive strength parallel to the grain or for modulus of elasticity, although a regression, significant at the 5 per cent. level of probability, had been found to be present for maximum tensile strength (3). It is unlikely that small differences in distance from pith would have much effect in a softwood and it was impossible to cut the various thicknesses at random distances from the pith in various logs because these were being used for other investigations as well as the one described here. In the selection of this material, any veneer containing compression wood, compression failures, knots, or other defects likely to affect the strength, was rejected.

The veneer, when dried, was glued into three-ply sheets 19½ in. square made with each of the following glues: (a) a casein glue, (b) a urea-formaldehyde glue, and (c) a phenol-formaldehyde film glue. In each sheet of plywood, the three veneers were closely matched. A total of 360 sheets was prepared: that is, 18 sheets from each log comprising two for each glue in each thickness.

Two sets of specimens were cut from each sheet, specimens parallel, at 45°, and perpendicular to the grain of the face veneer, being included. It was decided to test at four nominal moisture contents, namely 8, 12, 16, and 20 per cent. These values are based on the equilibrium moisture content of spruce (*Picea sitchensis* (Bon.) Carr.) slivers placed in the room whilst at a moisture content lower than that in equilibrium with the room, so that they would adsorb moisture. The four specimens available for each glue thickness and direction of grain in each log were allotted to the four moisture contents on the assumption that the two sheets made with the same glue and thickness of veneer from material from the same log were identical. The veneer in these had been carefully matched and the reason that they were not glued into one instead of two sheets was limitation of press capacity. However, the allocation of moisture contents between sheets was continually changed, in order to obviate any possibility of a systematic difference arising.

3. Moisture Conditioning Treatment

The test material was conditioned in humidity controlled rooms having a temperature in all cases of between 20° and 40°C. in order to avoid damage to the plywood. With the material for test at 8 per cent. moisture content, it was found necessary to use a temperature of 37°C. in order to reduce the relative humidity sufficiently, with the equipment available. For the remaining material the conditioning temperature was in the vicinity of 30°C. The conditions in these rooms were adjusted with the aid of thin slips of sitka spruce which were placed in the room at moisture contents of about 3 per cent. below the desired value. The humidity of the room was then gradually increased until the spruce slips reached the desired moisture content. These were then weighed every day to check the conditions and new ones, also reduced to about 3 per cent. below the desired moisture content, were occasionally introduced for check purposes. The slips varied by about ± 0.25 per cent. moisture content. The value was often constant to less than ± 0.1 per cent. for some days and then a fluctuation of ± 0.1 or ± 0.2 per cent. would occur. The variation between the values for two successive days was rarely more than this although occasional larger changes occurred, necessitating an adjustment of the room setting. When sudden fluctuations in moisture content occurred, new spruce slips were introduced as a check, in case those already there were in error due to hysteresis in the relative humidity-moisture content relationship. The apparent moisture content of the specimens when equilibrium had been reached was not, of course, the same as that of the spruce. This was particularly noticeable in the case of plywood glued with synthetic resin glues where the moisture content determined by oven-drying was some 2 to 3 per cent. below that of the spruce, the reason for which was probably loss in hygroscopicity due to the exposure of the plywood to elevated temperature during pressing.

4. Method of Test

All tests were carried out in a temperature range of 21°C. $\pm 1°$, the specimens being given time to reach this temperature before testing, since Kollman (5) and Sulzberger (10) have shown that considerable changes occur in the strength properties of wood and plywood due to changes of temperature.

Tension and compression tests were carried out, measurements of the elastic modulus being made only in the case of the tensile tests. The tests were carried out parallel, perpendicular, and at 45° to the grain of the outer veneers.

The tensile tests were carried out on specimens 12 inches long by 2 inches wide with a parallel portion 2½ inches long and 1 inch wide at the centre, and parallel portions of full width 2 inches long at the ends, the connecting radius being about 12 inches. The deformations were determined by means of a Gerrard extensometer measuring over a 2-in. gauge length, and load deformation curves were plotted. The testing procedure was generally in accordance with the Australian Standard Specifications (8).

The compression tests were carried out on packs of specimens 3½ inches long by 1 inch wide. The packs were made up into thicknesses of as near to an inch as possible in the case of 3/16 in. and ¼ in. plywood but this was not possible with the ⅛ inch plywood as the sheets

were not of sufficient size to cut sixteen pieces in each direction and the pack was only half an inch thick in this case. These packs were held in a special end-clamping apparatus developed by Thomas (12). A number of types had been tried but this one was found to be the most satisfactory. The same rate of loading was adopted as in the tensile tests.

5. Results of Tests

The results which have been obtained by dividing the maximum load by the total cross section of the specimen have been averaged and the averages plotted in Figs. 1 to 9. Figs. 1 to 3 show the results for modulus of elasticity as measured in the tensile tests, Figs. 4 to 6 for maximum tensile strength, and Figs. 7 to 9 for maximum crushing strength. Curves have been fitted by the method of least squares to the points shown, the significance of the regressions being indicated in Table 1 where the actual regression factors are given. The regression lines have been fitted using a logarithmic transformation as the data vary somewhat exponentially with moisture content. The use of transformations has been discussed in a report by Williams (13). The regression factors are the factors by which the strength at a given initial moisture content must be multiplied for each 1 per cent. decrease in moisture content to determine the strength at the decreased moisture content. For m per cent. decrease in moisture content it is therefore necessary to multiply by the mth power of the regression factor. That is, if r is the regression factor and S_n the strength or elastic modulus at a moisture content n, then the value $S_{(n+m)}$ at a moisture content $(n + m)$ is given by

$$\cdot S_n = S_{(n+m)} r^m$$

This is seen to be of the same form as Wilson's exponential formula (14) since on expressing the above formula in the exponential form it becomes $$S_n = S_{(n+m)} e^{m \log r}$$
which is identical with Wilson's formula when $\log 1/r$ is replaced by K. In the case of an increase in moisture content the strength value is divided instead of multiplied by the repression factor.

Since additive corrections cannot be made, Tables 2 to 4 have been computed giving the strength and elastic modulus at each integral percentage throughout the range of moisture content values covered by the experiment. In both figures and tables the direction of grain refers to that of the outer veneers relative to the loading axis of the specimen.

6. Discussion of Results

In some cases the results do not differ significantly from a linear relationship and an additive correction could be used. This can be seen from the fitted curves in Figs. 1 to 9. Such corrections have, however, not been given. Generally, the maximum crushing strength shows little difference in regression factors for different thicknesses or glues but the linear regressions on moisture content are always significant. Often the regression deviates significantly from the linear, especially for specimens cut at 45° to the direction of the grain. The moisture corrections themselves also vary considerably, for different grain directions, and somewhat for glues and occasionally thicknesses.

In the case of maximum tensile strength, the regression was not significant in eight of the 27 tests, whilst in two others the signficance was only at the 5 per cent. level of probability. Seven of the fifteen highly significant regressions also deviate significantly from linearity whilst two have no significant linear trend but have a quadratic term which is significant. In specimens tested parallel to the grain, the trends vary, the corrections being greatest for phenol-formaldehyde glue and least for casein. For specimens tested perpendicular to the grain, the regression is significant for all thicknesses with urea-formaldehyde glue, and these are the only ones for which the property increases with increasing moisture content. In no case is it significant for casein glue and only with ⅛-in. plywood for phenol-formaldehyde glue. For specimens tested at 45° to the grain, the corrections are rather variable and, in six cases, the regressions are curvilinear, the linear regression being non-significant for two of the cases where the quadratic term is significant.

All but one of the corrections for modulus of elasticity in tension parallel to the grain of the outer veneers, are significant, the regressions being linear. The corrections here do not differ significantly between types of glue or thicknesses although they do between directions of grain. For tension perpendicular to the grain of the outer veneers, the corrections do not differ significantly amongst themselves and so may be averaged, whilst those for tests at 45° differ significantly for different types of glue.

Where the corrections do not differ significantly between glues, thicknesses or direction of grain, average corrections could have been given. Strength figures, expressed as a percentage of the strength at 12 per cent. moisture content, have instead been tabulated for each integral percentage of moisture content throughout the range of the observations, for each glue, thickness and direction of grain.

In a recent unpublished report by Sulzberger (11), comparable values are to be found, although these apply only to one thickness of plywood, namely 3/16-in. three-ply, and to only one glue, namely urea-formaldehyde. For tensile strength, his results are a few per cent. higher parallel and perpendicular to the grain, but do not differ appreciably from the present ones at 45° to the grain. For modulus of elasticity, the same is true, except that at 45° to the grain a slight difference is apparent. The agreement is considerably closer for compressive strength parallel to the grain, although here again the results for tests at 45° to the grain differ slightly. This is illustrated in Fig. 10 for compressive strength.

7. Comparison with Solid Timber

No completely satisfactory comparison is possible between the variation of the strength of plywood and of solid timber with moisture content, as the values for the former vary so greatly amongst themselves. Expressed as regression factors, the moisture corrections for solid hoop pine are as follows (6):—

　　　1·012 for tension parallel to the grain;
　•1·047 for compression parallel to the grain;
　　　1·025 and 1·027 for compression perpendicular to the grain for the load applied to the tangential and radial faces respectively; and
　　　1·019 for modulus of elasticity (tensile test).

TABLE 1.—REGRESSION FACTORS OF STRENGTH PROPERTIES AND ELASTIC MODULUS OF HOOP PINE PLYWOOD ON MOISTURE CONTENT

Property	Direction of Grain of Face Veneer	Phenol-Formaldehyde Glue			Urea-Formaldehyde Glue			Casein Glue		
		⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood	⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood	⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood
Maximum Crushing Strength	Parallel	1·058**	1·082**	1·060**	1·063**	1·059**C.	1·053**	1·057**	1·059**	1·057**
	Perpendicular	1·060**	1·068**	1·087**	1·062**	1·071**C.	1·059**	1·062**	1·062**	1·062**C.
	45°	1·054**C.	1·048**C.	1·045**	1·042**	1·048**C.	1·023**	1·047**C.	1·048**C.	1·040**C.
Maximum Tensile Strength	Parallel	1·026**C.	1·016**C.	1·016 N.S.	1·005 N.S.	1·012**	1·020**C.	1·001 N.S.	1·012**	1·017**
	Perpendicular	1·014*	1·005 N.S.	1·007 N.S.	·975**	·984**	·978**	1·001 N.S.	1·001 N.S.	1·004 N.S.
	45°	1·017**	1·012**	1·004 N.S. C.	1·011**C.	1·005*	·998 N.S. C.	1·016**C.	1·017**C.	1·008**C.
Modulus of Elasticity	Parallel	1·026**	1·011**	1·018**	1·013**	1·010**	1·026**	1·016**	1·015**	1·007 N.S.
	Perpendicular	1·021**	1·013*	1·008 N.S.	1·009**	1·015**	1·011 N.S.	1·013**	1·015**	1·013**
	45°	1·040**	1·045**C.	1·043**	1·036**	1·030**C.	1·022**	1·040**	1·028**C.	1·038**

** Significant at the 1 per cent. level of probability.
* Significant at the 5 per cent. level of probability.
C. Regression departs significantly from the linear at the 1 per cent. level of probability.
N.S. No significant linear regression.
For an increase of 1 per cent. moisture content, divide by the regression factor.

TABLE 2.—TENSILE MODULUS OF ELASTICITY OF HOOP PINE PLYWOOD AT VARIOUS MOISTURE CONTENTS
(Expressed as a percentage of the value at 12 per cent. moisture content)

Glue	Percentage Moisture Content	Parallel to the Grain			Perpendicular to the Grain			At 45° to the Grain		
		¼ in Plywood	⅜ in Plywood	½ in Plywood	¼ in Plywood	⅜ in Plywood	½ in Plywood	¼ in Plywood	⅜ in Plywood	½ in Plywood
Phenol-formaldehyde	6	116	107	111	113	108	105	126	130	129
	7	114	106	109	111	107	104	122	124	124
	8	111	104	108	109	105	103	117	119	118
	9	108	103	106	108	104	102	113	114	114
	10	105	102	104	104	103	102	108	109	109
	11	103	101	102	102	101	101	104	104	104
	12	100	100	100	100	100	100	100	100	100
	13	97	99	99	98	99	99	98	96	96
	14	95	98	97	96	98	98	92	92	92
	15	93	97	95	94	96	98	89	88	88
	16	90	95	93	92	95	97	85	84	85
	17	88	95	92	90	94	96	82	80	81
Urea-formaldehyde	6	108	106	117	105	109	107	124	119	114
	7	107	105	114	104	108	106	119	116	112
	8	105	104	111	104	106	104	115	112	109
	9	104	103	108	103	105	103	111	109	107
	10	103	102	105	102	103	102	107	106	105
	11	101	101	103	101	102	101	103	103	102
	12	100	100	100	100	100	100	100	100	100
	13	99	99	97	99	99	99	97	97	98
	14	98	98	95	98	97	98	93	94	96
	15	96	97	93	97	96	97	90	92	94
	16	95	96	90	96	94	96	87	89	92
	17	94	95	88	96	93	95	84	86	90
	18	93	94	86	95	92	94	81	84	88
	19	92	93	84	94	90	93	78	81	86
Casein	8	106	106	103	106	106	105	117	112	116
	9	105	104	102	104	105	104	113	109	112
	10	103	103	102	103	103	103	108	106	108
	11	101	101	101	101	102	101	104	103	104
	12	100	100	100	100	100	100	100	100	100
	13	98	99	99	98	98	99	96	98	96
	14	97	96	98	96	97	98	92	95	93
	15	95	95	98	96	96	96	89	92	90
	16	93	94	97	95	94	95	86	90	88
	17	92	92	96	94	93	94	82	87	83
	18	91	92	96	93	92	93	79	85	80
	19	89	90	95	91	90	92	76	82	77
	20	88	88	94	90	88	90	73	80	74

TABLE 3.—MAXIMUM TENSILE STRENGH OF HOOP PINE PLYWOOD AT VARIOUS MOISTURE CONTENTS
(Expressed as a percentage of the value at 12 per cent. moisture content)

Glue	Percentage Moisture Content	Tension Parallel to the Grain			Tension Perpendicular to the Grain			Tension at 45° to the Grain		
		⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood	⅜ in. Plywood
Phenol-formaldehyde	6	117	110	111	109	103	104	111	108	103
	7	113	108	109	107	103	104	109	106	102
	8	111	106	107	106	102	103	107	105	102
	9	108	105	105	104	102	102	105	104	101
	10	105	103	104	103	101	101	103	103	101
	11	103	102	102	101	100	101	102	101	100
	12	100	100	100	100	100	100	100	100	100
	13	98	98	99	99	99	99	99	99	99
	14	95	96	97	97	98	98	97	98	99
	15	92	96	96	96	98	98	95	97	98
	16	90	94	94	94	98	97	93	96	98
	17	88	92	92	93	97	96	92	94	98
Urea-formaldehyde	6	103	107	112	86	91	88	107	103	98
	7	103	106	109	88	92	90	105	102	99
	8	102	105	108	90	94	91	104	102	99
	9	102	104	108	93	95	94	103	101	100
	10	101	103	104	95	97	96	102	101	100
	11	101	101	102	98	98	98	101	100	100
	12	100	100	100	100	100	100	100	100	100
	13	99	99	98	103	102	102	99	99	100
	14	99	97	96	105	103	104	98	99	101
	15	98	96	94	108	105	107	98	98	101
	16	98	95	90	111	107	109	97	98	101
	17	97	93	88	114	108	112	97	97	101
	18	97	93	88	117	110	114	97	97	101
	19	96	92	87	120	112	118	96	96	101
Casein	8	101	105	107	100	100	102	107	107	103
	9	101	104	105	100	100	101	105	105	102
	10	100	102	103	100	100	101	103	103	102
	11	100	101	101	100	100	100	101	102	101
	12	100	100	100	100	100	100	100	100	100
	13	100	99	98	100	100	100	98	98	99
	14	100	97	97	100	100	99	97	97	98
	15	100	96	95	99	99	99	95	95	98
	16	100	95	94	99	99	98	94	94	97
	17	100	94	92	99	99	98	92	92	96
	18	100	93	90	99	99	98	91	90	95
	19	99	92	89	99	99	97	89	89	94
	20	99	91	87	99	99	97	88	88	94

TABLE 4.—MAXIMUM CRUSHING STRENGTH OF HOOP PINE PLYWOOD AT VARIOUS MOISTURE CONTENTS
(Expressed as a percentage of the value at 12 per cent. moisture content)

Glue	Percentage Moisture Content	Compression Parallel to the Grain			Compression Perpendicular to the Grain			Compression at 45° to the Grain		
		⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood	⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood	⅛ in. Plywood	¼ in. Plywood	⅜ in. Plywood
Phenol-formaldehyde	6	140	144	142	142	148	148	137	131	130
	7	132	135	134	134	139	139	130	125	124
	8	125	127	126	126	130	130	122	120	119
	9	118	120	119	119	122	122	117	114	114
	10	112	113	112	112	114	114	111	109	109
	11	106	106	106	106	107	107	106	105	104
	12	100	100	100	100	100	100	100	100	100
	13	95	94	94	94	94	94	95	96	96
	14	89	89	89	89	88	88	90	91	91
	15	84	83	84	84	82	82	86	87	87
	16	80	79	79	79	77	77	81	84	84
	17	75	74	75	75	72	72	77	80	80
Urea-formaldehyde	6	145	141	136	144	151	141	128	131	115
	7	136	133	129	135	141	133	123	125	112
	8	128	126	123	127	131	126	118	120	110
	9	120	119	117	120	123	119	113	115	107
	10	113	112	111	113	115	112	108	110	105
	11	106	106	105	108	107	106	104	105	102
	12	100	100	100	100	100	100	100	100	100
	13	94	94	95	94	93	94	96	96	98
	14	88	89	90	89	87	89	92	91	96
	15	83	84	86	83	82	84	88	88	93
	16	78	79	81	79	76	80	85	84	92
	17	74	75	77	74	71	75	81	80	89
	18	69	71	73	70	66	71	78	76	87
	19	65	67	70	66	62	67	75	73	85
Casein	8	125	126	125	127	127	127	120	120	...
	9	118	119	118	120	120	120	115	115	112
	10	112	113	112	114	113	113	110	110	108
	11	106	106	106	106	106	106	105	105	104
	12	100	100	100	100	100	100	100	100	100
	13	95	94	95	94	94	94	95	96	96
	14	89	89	90	89	89	89	91	92	92
	15	85	84	85	84	83	83	87	88	89
	16	80	80	80	78	79	79	83	84	86
	17	76	75	76	74	74	74	80	80	82
	18	72	71	72	70	70	70	76	76	79
	19	68	67	68	66	66	66	72	73	76

Fig.1 Variation of Modulus of Elasticity with Moisture Content for Phenol-Formaldehyde bonded Plywood.

Fig.2 Variation of Modulus of Elasticity with Moisture Content for Urea-Formaldehyde bonded Plywood.

Fig 3 Variation of Modulus of Elasticity with Moisture Content for Casein bonded Plywood

Fig 4 Variation of Maximum Tensile Strength with Moisture Content for Phenol-Formaldehyde bonded Plywood

Key
○ ¼ Plywood
● ⅜ Plywood
● ⅜ Plywood

45 Degrees to the Grain
Parallel to the Grain
Perpendicular to the Grain

M of E 1000 lb/sq in
M of E 1000 lb/sq in
M of E 1000 lb/sq in
MTS lb/sq in
MTS lb/sq in
MTS lb/sq in

Moisture Content %

Fig. 5. Variation of Maximum Tensile Strength with Moisture Content for Urea-Formaldehyde bonded Plywood.

Fig. 6. Variation of Maximum Tensile Strength with Moisture Content for Casein bonded Plywood.
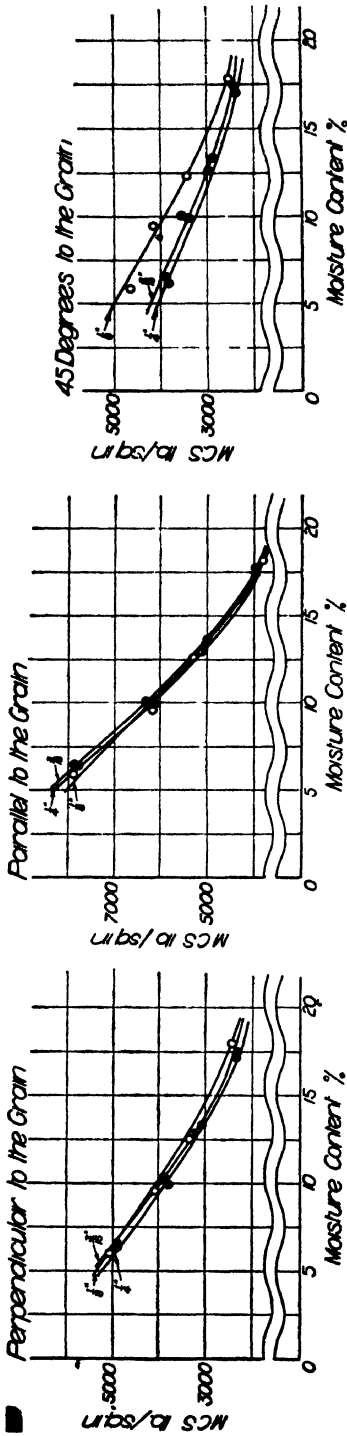
Fig 7 Variation of Maximum Crushing Strength with Moisture Content for Phenol-Formaldehyde bonded Plywood.
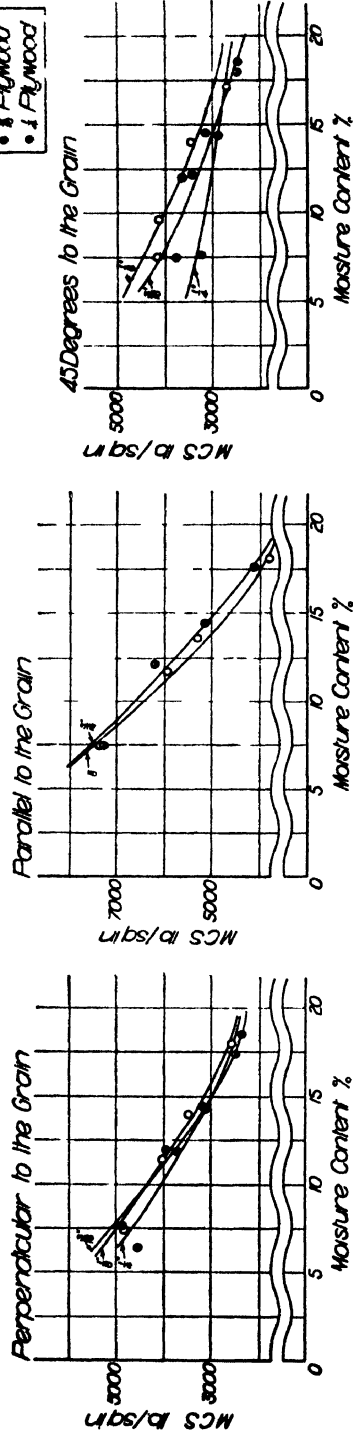
Fig 8 Variation of Maximum Crushing Strength with Moisture Content for Urea-Formaldehyde bonded Plywood
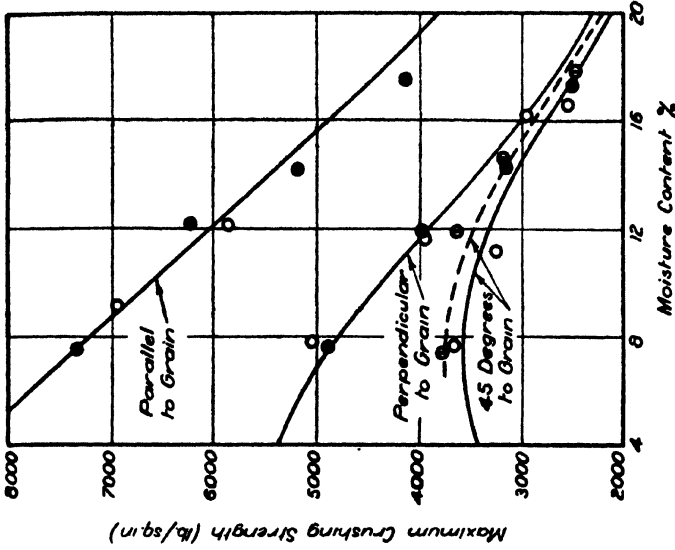
Fig.10. Comparison of Results Obtained by Sulzberger and the Author for the Effect of Moisture Content on Maximum Crushing Strength of Hoop Pine Plywood at 20°C.
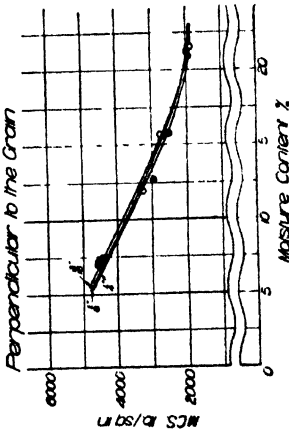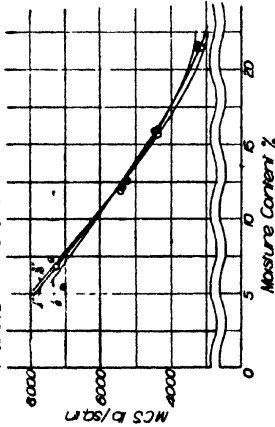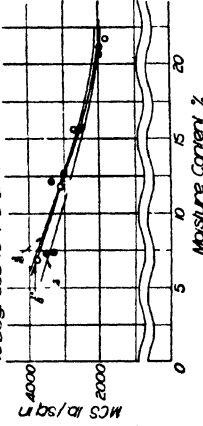
Fig 9 Variation of Maximum Crushing Strength with Moisture Content for Casein bonded Plywood.

The above regression factors for solid timber were determined by expressing the additive moisture corrections as a fraction of the mean in each case and adding the results to unity, in agreement with the convention adopted here, since these regression coefficients for solid timber are all negative.

The corrections for plywood are of the same order of magnitude as those for solid timber where rough comparisons can be made, except for maximum crushing strength, parallel and perpendicular to the grain. Here the regression factors are in general somewhat higher than those for solid timber either parallel or perpendicular to the grain. However, in view of the fact that there is no great difference between the regression coefficients for plywood and solid timber, values predicted from corrections for solid timber could be used for plywood of other species until actual corrections for plywood of those species are available. This general agreement has also been observed for Douglas fir and Sitka spruce (1).

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The Extraction of Lignin from *Eucalyptus regnans* F.v.M. by Means of Methanol

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Summary

A survey of possible methods of extracting native lignin from immature *Eucalyptus regnans* F.v.M. led to the conclusion that one of the most promising solvents was methanol. Treatment with this solvent for 24 hours at 150°C removed a little more than one quarter of the lignin, estimated as Klason lignin, present in a specimen of *E. regnans* sapwood sawdust. Treatment for 148 hours, using fresh methanol every sixteen hours, removed about 70 per cent. of the original lignin estimated as Klason lignin. Evidence was obtained to show that the methanol extraction of wood is essentially a mild acid hydrolysis accompanied by the solvent action of methanol. During the process some of the extracted lignin was degraded to materials soluble in water and ether. The remainder could be precipitated as a buff powder which gave the colour reactions of native lignin and which was soluble in bisulphite solutions.

I. Introduction

Lignin has not been obtained as a pure substance and has therefore not been characterized by definite physical constants and chemical properties, nor is there any concise definition of lignin. It is known, however, to be a biological material readily denatured by extraction processes. When obtained as a by-product from the wood pulp industry it is extensively degraded and differs widely in its properties from the lignin as it occurs in Wood. Methods of isolation involving the destruction of the cellulose by concentrated acids give lignins which are highly polymerized and non-reactive. Klason lignin, isolated from wood or pulp by means of 72 per cent. sulphuric acid, is widely recognized provisionally as a measure of the lignin content.

Aronovsky and Gortner (1936) pulped aspen wood with aqueous alcohols and found that butanol was the most effective, reducing the lignin content of the pulp to 3 per cent. It was suggested that the ligneous material so obtained was less denatured than lignins obtained by inorganic reagents. Bailey (1940) found that the lignin was almost completely removed from aspen wood by cooking with butanol and water. The removal was greatly accelerated by the presence of 2 per cent. sodium hydroxide, which he considered to act as a catalyst. He noticed that, with neutral butanol cooks, the cooking liquor became acid.

Charbonnier (1942) found that neutral butanol and water extracted only a small part of the lignin from aspen wood. He was thus unable to confirm the observations of Bailey on the pulping qualities of butanol. He found it necessary to add sodium hydroxide, as Bailey had in most of his experiments, and considered that the lignin thus obtained was not a native lignin but a typical alkali lignin.

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Hewson, McCarthy, and Hibbert (1941) were able to remove somewhat less than half of the lignin from maple wood by extraction with ethanol for four hours at 200°C. This produced, besides ethanol lignin, " lignin tar " and some distillable oils. They considered that the drastic conditions necessary indicated that the lignin units were in combination in the wood as ethanol-insoluble aggregates.

Overbeck and Müller (1942) cooked various woods with water at temperatures ranging from 130° to 165°C. They found that some of the wood substance was dissolved and that part of the lignin became soluble in alcohol. They also noticed that the cooking liquor became acid and concluded that the process was essentially an acid hydrolysis. The residual lignin in the wood was somewhat more resistant to sulphite cooking than the original lignin.

Katzen, Sawyer, and Othmer (1945) showed that lignin solubility decreased with increase in the number of carbon atoms in monohydric alcohols.

Brauns (1939) extracted spruce wood with water and ether and then with alcohol at room temperature. On concentrating the alcoholic solution and pouring into distilled water, a creamy precipitate was obtained which gave all the reactions of native lignin. It constituted, however, only a small part of the total lignin in the wood.

No work has yet been reported on the native lignin of eucalypts.

2. Outline of Experimental Procedure

(i) *Preparation of the Wood Sample*

Immature trees of *Eucalyptus regnans* F.v.M. ranging in age from six to ten years and free from truewood, were selected. Truewood was avoided because it was considered that the more severe extraction conditions necessary for the removal of the extraneous materials might remove some lignin. The logs were cut into short lengths, the bark removed, gum veins, pockets, and knots sawn out, and the remaining clear wood reduced to sawdust. In the first experiments, the sawdust was washed with water and then dried by repeated washing at room temperature with dry methanol. Later, it was found that the extraction of the lignin was not appreciably affected by oven-drying the sawdust. For most of the experiments, therefore, the wood was washed three times with cold water, three times with hot water, air-dried and then oven-dried. Alcohol treatment was not given as it was considered that it might remove some of the native lignin described by Brauns. Later experiments showed that the amount of lignin thus removed was negligible.

(ii) *Preliminary Extraction Experiments*

Some pilot experiments with lower monohydric alcohols showed that methanol was the most effective lignin extractant. As the introduction of alkoxyl groups other than methoxyl would complicate the methoxyl determination, and since methanol could very much more easily be evaporated from the wood than could a higher alcohol, it was decided to investigate thoroughly the action of methanol. The drying of methanol-wet wood was accordingly investigated. Following this, extractions were carried out at temperatures ranging from 60° to

160°C. with the following solvents: (i) methanol, (ii) methanol plus 0·4 per cent. water, (iii) methanol plus 2·0 per cent. dry hydrogen chloride, (iv) methanol plus 0·4 per cent. water plus 2·0 per cent. dry hydrogen chloride. Under the same conditions, increasing amounts of lignin were removed by the solvents in the order enumerated. The time factor was also important, more lignin being extracted with longer periods of time up to 160 hours. Methanol at 160°C. caused the residual wood to have a slightly charred appearance, whereas methanol containing 0·4 per cent. water and methanol containing 2·0 per cent. hydrogen chloride produced wood residues with colours close to that of the original sawdust. The methanol-hydrogen chloride, besides removing more lignin than did dry methanol, also dissolved a considerable amount of the carbohydrate material. The solvents containing water produced a dark red lignin, whereas that obtained with dry solvent was buff coloured. Since methanol appeared to dissolve the lignin more specifically than did the other solvents, its action was further investigated.

(iii) *Methanol Extractions*

Extractions of oven-dry wood with methanol at 150°C were carried out for various times. Mild steel bombs containing 150 ml. of methanol, 6·0 g. of dry sawdust, and with about 20 ml. of air space, were tumbled in a thermostatically controlled oil bath for the required time. Later, extractions of 1 kg. of sawdust with 12 l. of methanol were carried out in a mechanically-stirred, stainless steel autoclave. Each charge was subjected to one 4-hour and then nine 16-hour extractions, with fresh methanol used for each extraction. The amounts of Klason lignin and ester in the methanol were estimated for each extraction.

The total material removed was estimated from the loss of weight of the wood sample after extraction, drying and weighing. The total weight of extract was determined by evaporating an aliquot portion of the methanol solution to dryness on the water bath. In later experiments the extract was analysed for precipitated lignin, Klason lignin and esters. The wood residues were analysed for holocellulose, Cross and Bevan cellulose, pentosans, Klason lignin, and methoxyl. The methanol solutions were concentrated and methods of precipitating the lignin were investigated. The existence of water-soluble materials giving lignin reactions was demonstrated. The precipitated lignins and Klason lignins from the residual woods were analysed for methoxyl and the methoxyl not in Klason lignin was calculated for the original wood and for the methanol extracted woods.

(iv) *Methanol-HCl Extractions*

After the methanol extraction, the residual wood was treated for different periods with boiling methanol containing 2·0 per cent. dry hydrogen chloride. The residues were analysed as before.

(v) *Investigation of the Action of Methanol*

The action of methanol on acetic acid and on certain sugars in the presence of acetic acid was investigated. Methanol recovered from the large scale extractions was examined for the presence of acids and esters.

3. Results and Discussions

The drying of alcohol-wet wood powders proved to be difficult. This was to be expected after the observation of Cohen (1936) that wood powders retain a certain amount of ethanol. A one gram water-wet sample of *E. regnans* wood meal took about 1½ hours to dry at 100°C. A methanol-wet sample of the same size took about three times as long at the same temperature, notwithstanding the higher volatility of the methanol. This difficulty was more marked with ethanol and very much more with butanol. During the drying of saw-dust wet with methanol a distinct odour of formaldehyde was noted. Exhaustive washing of oven-dried methanol-extracted wood samples with ether did not appreciably reduce the methoxyl content; it appears from this that the methanol may not have been retained as such.

The amounts of material removed by methanol extraction under different conditions are shown in Tables 1, 2, and 3. It appears from the results in Table 1 that the total material removed was greater than

TABLE 1.—RESULTS OF EXTRACTION OF 60-80 MESH *E. regnans* WITH DRY METHANOL FOR 24 HOURS AT DIFFERENT TEMPERATURES

Temperature (Deg. C)	Total Material Removed*	Klason Lignin Removed*
60	0·9	2·5
110	3·5	3·2
160	21·0	15·5

* All results expressed as percentages of the original wood. Original wood 21·9 per cent. Klason lignin.

the total amount of lignin removed, and from Table 2 that the total amount of material removed from the wood was greater than the total weight of extract obtained on evaporation of the methanol. This was in turn greater than the total Klason lignin extracted, which again was greater than the amount of lignin recovered by precipitation. The excess total material removed over the weight of extract became pro-gressively greater with time and may have been due to loss of volatile materials during evaporation. The result at 60°C, given in Table 1, is apparently inconsistent in that the total extractives were less than the amount of Klason lignin lost. This is probably due, however, to the very small amount of material extracted and therefore the large influence of the error of drying methanol-wet wood.

In the first experiments, precipitation of the lignin was carried out by pouring the concentrated methanol solution into distilled water; this caused some of the lignin to go into colloidal solution, only a variable and indefinite fraction being precipitated. An electrophoresis experiment showed that the lignin was present as a negatively-charged colloid. It was found that the water-insoluble lignin could be pre-cipitated by making the solution alkaline with sodium hydroxide and then adding hydrochloric acid until the solution was decinormal in acid. This procedure brought down the lignin as a buff-coloured precipitate.

It could also be precipitated by the addition of salts. The filtrate from the precipitation of the lignin reduced Fehling's solution only very feebly, even after boiling with hydrochloric acid, and it was also negative to Molisch's reagent. Lignin reactions, however, both with chlorine and sodium sulphite and with phloroglucinol, were strongly positive. Repeated extraction of this filtrate with benzene or ether finally made the aqueous layer negative to the phloroglucinol test and only feebly positive to the chlorine and sodium sulphite test. On evaporation of the combined benzene or ether extracts, a sticky resinous material giving strong lignin reactions was obtained.

It can be seen from Table 2 that the rate of removal of lignin estimated as Klason lignin decreased rapidly with time. After treatment for four days the water-solubility of the wood meal had increased from 1·0 to 7·2 per cent., and the alkali-solubility had increased from 9·7 to 17·3 per cent. of the weight of the original wood.

TABLE 2.—RESULTS OF EXTRACTION OF OVEN-DRY *E. regnans* SAWDUST WITH DRY METHANOL AT 150°C. FOR VARIOUS TIMES

Duration of Extraction	Total Material Removed	Weight of Extract	Weight of Precipitated Lignin	Klason Lignin Extracted
4 hours	5·4	5·3	4·0	2·8
1 day	14·1	13·1	8·3	9·3
2 days	19·4	17·7	11·2	11·5
4 days	24·5	22·4	9·0	14·6
6 days	24·2	20·9	10·2	14·6

All results expressed as percentages of the original wood. Original wood 22·6 per cent. Klason lignin.

The results of the extraction of 1 kg. of sawdust are shown in Table 3. The yield of Klason lignin from the extract was determined by treating the total solids, obtained on evaporation of the methanol, according to the Division of Forest Products standard method for lignin determination. It can be seen that the ratio of Klason lignin to methyl acetate, excluding the ratio for the preliminary four-hour extraction, increased from four to thirteen between the first and ninth sixteen-hour extractions.

The methoxyl contents of precipitated methanol lignins, of Klason lignin and of the residual wood are shown in Table 4. The methoxyl content of the methanol lignin (column 2) recovered from extractions lasting longer than four hours was practically constant at a value considerably higher than that for the Klason lignin either from the original wood or from any of the residual woods. The methoxyl in Klason lignin was calculated as a percentage of the original wood thus: after one day the Klason lignin content of the residual wood was 13·3 per cent. of the original wood. Hence (22·6 — 13·3) = 9·3 per cent. of Klason lignin had been extracted. The residual Klason lignin contained 22·8 per cent. methoxyl and therefore the methoxyl in lignin as a percentage of the wood was 22·8 per cent. of 13·3 = 3·03 per cent., whereas the total methoxyl content of the wood was 5·62 per cent., and therefore methoxyl not in Klason lignin constituted 2·59 per cent. of the wood.

The analyses of the original wood and of the wood after methanol extraction for different periods of time are shown in Table 5. Whereas, in the original wood, the sum of the percentages of lignin and holo-cellulose was close to 100, this was not so for the extracted woods,

TABLE 3.—LIGNIN AND ESTERS PRODUCED BY SUCCESSIVE METHANOL
EXTRACTIONS OF *E. regnans* SAWDUST

Extraction Time	Total Solids in Extract (a)	Klason Lignin in Extract (b)	Ratio a/b	Pre-cipitable Lignin in Extract (c)	Esters as Methyl Acetate (d)	Ratio b/d	Methoxyl Content of Pre-cipitated Lignin
4 hr.	2·95	2·32	1·27	1·15	0·25	9·3	18·7
1st 16 hr. ..	4·42	3·19	1·38	1·42	0·74	4·3	24·1
2nd 16 hr. ..	3·67	2·77	1·32	1·31	0·66	4·2	26·0
3rd 16 hr. ..	2·93	2·21	1·33	1·13	0·41	5·4	26·0
4th 16 hr. ..	2·28	1·70	1·34	0·94	0·25	6·8	25·6
5th 16 hr. .	1·68	1·25	1·34	0·73	0·14	8·9	26·7
6th 16 hr. ..	1·33	0·94	1·41	0·55	0·10	9·4	26·3
7th 16 hr. ..	1·10	0·74	1·49	0·42	0·08	9·3	25·4
8th 16 hr. ..	0·95	0·58	1·64	0·35	0·06	9·7	26·2
9th 16 hr. ..	0·85	0·53	1·60	0·30	0·04	13·2	26·5
Total ..	..	16·23	..	8·30	2·73	..	..

a, b, c, and *d*—percentage of original wood.

the discrepancy becoming more marked after the longer period of treatment. This must be due to portion of the carbohydrate fraction becoming soluble in the reagents used. Only two to four chlorinations and subsequent ammoniacal alcohol treatments were required to delignify the methanol-extracted samples in the holocellulose determination, whereas the original wood required eight. This, combined with the large increase in water-soluble material mentioned above, shows clearly that the carbohydrates of the wood were considerably modified by the methanol treatment.

TABLE 4.—METHOXYL CONTENTS OF ISOLATED LIGNINS AND
RESIDUAL WOODS

Duration of Extraction	Precipitated Methanol Lignin (Percentage Lignin)	Klason Lignin		Total Methoxyl in Wood (Percentage)	Methoxyl not in Klason Lignin (Percentage Original Wood)
		Percentage Lignin	Percentage Original Wood		
0 (Original wood)	..	22·6	5·11	7·48	2·37
4 hours ..	26·5	23·6	4·67	6·97	2·30
1 day ..	28·3	22·8	3·03	5·62	2·59
2 days ..	28·6	21·9	2·43	4·55	2·12
4 days ..	28·6	21·7	1·73	3·54	1·81
6 days ..	28·7	22·0	1·76	3·54	1·78

Original wood 22·6 per cent. Klason lignin.

The results of the methanol-HCl extractions of the methanol-extracted wood residues are shown in Table 6. It is apparent that a further considerable quantity of lignin was rapidly taken into solution,

but that extension of the extraction time beyond four hours did not extract much more. In this case the ratio of the total amount of material extracted to the amount of recovered lignin (about 4 : 1) is greater than the corresponding ratio (about $2\frac{1}{2}$: 1) for the methanol extraction. There remained in the wood a third fraction of the lignin which resisted even the methanol-HCl treatment.

TABLE 5.—ANALYSIS OF METHANOL EXTRACTED *E. regnans*
(*Per cent. oven-dry sample weight.*)

Duration of Extraction	Lignin	Holocellulose	Lignin + Holocellulose
0 (Original wood)	24·0	75·4	99·4
4 hours	21·3	73·8	95·1
4 hr. + 16 hr. (1)	17·4	70·3	87·7
4 hr. + 16 hr. + methanol-HCl extraction (2)	9·2	72·3	81·5

(1) After extraction for four hours again extracted for sixteen hours with fresh solvent.
(2) Residue from (1) given methanol-HCl extraction for four hours.

A wood sample and the residue from the same wood after 72 hours of methanol extraction were analysed and the results are given in Table 7. It can be seen that the holocellulose content of the residual wood was only 8 per cent. higher than the Cross and Bevan cellulose content, whereas in the original wood it was 22 per cent. higher. There was only a small loss of Cross and Bevan cellulose and more than half of this was accounted for by loss of xylan. Thus the true cellulose remained practically unaffected by the methanol extraction, whereas the hemicelluloses were largely affected so as to render them soluble in the holocellulose reagents. These results constitute yet further indication that it is not possible to remove lignin from the wood without affecting the carbohydrate portion, chiefly the hemicelluloses, and that rupture of the ligno-cellulose bond involves liberation of some of the hemicelluloses.

TABLE 6.—RESULTS OF METHANOL-HYDROCHLORIC ACID EXTRACTION OF METHANOL-EXTRACTED WOOD

of Extraction	Amount Extracted	Weight of Precipitated Lignin	Klason Lignin Content of Wood Residue
0	..	..	10·30
5 mins.	4·7	1·50	7·15
4 hours	16·1	4·45	4·81
24 hours	20·2	4·77	4·10

All results expressed as percentages of original wood

The methanol recovered from successive sixteen-hour extractions of 1 kg. of wood was found to contain esters (estimated by saponification and calculated as methyl acetate) in amounts shown in Table 3. Total esters produced during nine successive sixteen-hour extractions

amounted to 2·73 per cent. of the weight of the original wood. The original wood contained 3·2 per cent. acetyl and the last residue 1·3 per cent.

Lignin is not extractable by cold methanol but, once the methanol lignin is obtained, it is freely soluble in cold methanol. This is a clear indication that this material is derived by the breakdown of some larger molecule such as a lignin carbohydrate compound.

When 1 g. of acetic acid was heated in 150 ml. of methanol at 150°C. for 16 hours, it was found to be almost completely esterified. This does not exclude the possibility that acid conditions obtained throughout the extraction, because acidity could be maintained by slow liberation of acid from the wood. It can be seen from Table 3 that the ratio of Klason lignin extracted to total material extracted was roughly constant. From the ratio of Klason lignin in extract to methyl acetate formed, it appears that the formation of methyl acetate took place concurrently with the lignin removal, greater amounts of the ester being formed per unit amount of lignin as the extraction proceeded. This may be interpreted as showing that the methanol extraction is a mild acid hydrolysis. From the fact that the non-lignin extractives became larger in amount and reducing sugars were detectable in the liquor, it may be concluded that the further solution of lignin accomplished by methanolysis was accompanied by the hydrolysis of carbohydrate material. It appears, therefore, that the weakly acid methanol extraction opens the ligno-cellulose bond and removes lignin more specifically than do acid extractions.

TABLE 7.--ANALYSIS OF ORIGINAL WOOD AND WOOD AFTER 72 HOURS METHANOL EXTRACTION

—	Lignin	Holo-cellulose	Cross and Bevan Cellulose	Xylan in Wood	Xylan in Holo-cellulose	Xylan in Cross and Bevan Cellulose
Original wood	22·6	81·0	59·1	17·4	16·8	11·0
Extracted wood (19·8 per cent. removed) ..	10·6	63·0	55·0	16·4	11·7	8·4

All results expressed as percentages of original wood.

4. Properties of Lignin Isolated by Methanol Extraction

The isolated lignin was a buff-coloured powder which could be dissolved in methanol and re-precipitated by pouring into a large quantity of cold ether, when it came out as a flesh-coloured powder which rapidly darkened to brown in air. It was freely soluble in sodium hydroxide to give a brown solution, and it could be re-precipitated from this solution by acid. It was soluble in alcohol, chloroform, acetone, dioxan and hot cyclohexanol, but was very sparingly soluble in cold cyclohexanol. It was insoluble in benzene,

ether and petroleum ether. Ultramicroscopic examinations of the methanol and sodium hydroxide solutions showed that they were true and not colloidal solutions. These solubility properties distinguish it sharply from acid lignins such as Klason lignin.

The isolated lignin gave the typical pored-wood lignin colour reactions, namely, cherry red when treated with chlorine water followed by sodium sulphite, and colours with phenols and aromatic amines. With phloroglucinol it was a typical deep purple and with aniline a rich yellow. It gave no colour with ferric chloride.

By taking the temperature at which the lignin became sticky in the tube, fairly definite softening points could be obtained for the various samples of isolated lignin. Values varied between 110°C. and 132°C.

After heating for sixteen hours at 120°C. in sodium bisulphite solution containing 5 per cent. total sulphur dioxide, or in sulphite cooking liquor containing 6·1 per cent. total and 1·1 per cent. combined sulphur dioxide, the isolated methanol lignin was completely dissolved. Under the same conditions Klason lignin from the same wood was insoluble.

These properties of methanol lignin are therefore properties of the lignin in the wood which are not shared by acid lignins.

Repeated heating of the water-insoluble lignin in methanol at 150°C. resulted in its degradation to water-soluble materials. It appears therefore that the water-soluble material arose from breakdown of lignin during the extraction and that a percolation method would yield a less degraded lignin. The methanol extraction appears to be relatively free from the polymerization actions observed by Hewson and Hibbert (1943) since no methanol-insoluble residue was produced, as there was during methanol-HCl treatments of methanol lignin. Nevertheless prolonged methanol extraction did not completely delignify the wood. On boiling an aqueous solution of the water-soluble lignin with 3 per cent. sulphuric acid, a reddish-brown precipitate resembling Klason lignin was produced.

Methanol lignin, on treatment with dry hydrogen chloride in methanol, was changed to a black material soluble in sodium hydroxide but insoluble in the sodium bisulphite solution. Part of it was also precipitated as a black residue insoluble in methanol. Methylation with diazomethane gave a cream-coloured powder insoluble in sodium hydroxide.

These results are qualitatively the same as those obtained by Brauns with his spruce native lignin. However, the methoxyl content of methanol lignin from *E. regnans* (25-28 per cent.) was much higher than that of Brauns' native lignin (14·8 per cent.), and higher also than that of Klason lignin from *E. regnans* (22 per cent.).

Brauns and Brown (1938) concluded that sulphite solubility was dependent upon an easily methylated hydroxyl group. Since the isolated lignin was soluble in bisulphite it may be concluded that this group remained unmethylated.

Mackney (1940) showed that the methoxyl in holocellulose plus methoxyl in Klason lignin did not account for the total methoxyl in the wood of *E. regnans*. Increasingly drastic alkaline treatments of the wood caused loss of methoxyl from the holocellulose, but the unaccounted methoxyl remained constant. It appears therefore that this unaccounted methoxyl was originally attached to the lignin but was removed during the Klason treatment. The methoxyl in holocellulose was 1·2 per cent. and the unaccounted methoxyl 1·1 per cent., a total of 2·3 per cent. methoxyl not in Klason lignin. This is in agreement with the value of 2·37 per cent. methoxyl not in Klason lignin given in Table 4, and which is an entirely independent determination.

If it is assumed that all methoxyl not in holocellulose is associated with the native lignin, then we may calculate the expected methoxyl content of the native lignin of *E. regnans* from the data in Table 4 and Mackney's figure for the non-lignin methoxyl. The total methoxyl in the wood was 7·48 per cent. and therefore the methoxyl associated with the lignin was 6·28 per cent. The Klason lignin content of the wood was 22·6 per cent. Since methanol lignin yielded only 90 per cent. of Klason lignin, it must be assumed for purposes of calculation that this represents 24·9 per cent. of methanol lignin in the wood. The methoxyl content of the native lignin would therefore be $\dfrac{628}{24\cdot9} = 25\cdot2$ per cent. Similarly using Mackney's figure for methoxyl not in Klason lignin (2·3 per cent.) the methoxyl in Klason lignin would then be 5·18 per cent. of the wood, which contained 22·6 per cent. Klason lignin, and the expected methoxyl content of the Klason lignin is therefore $\dfrac{518}{22\cdot6} = 22\cdot9$ per cent.

It is recognized that these calculations are open to objections in that it is not known with any degree of certainty that the Klason lignin really does represent all the lignin and nothing but the lignin; furthermore, the calculations apply to the whole of the lignin in the wood, whereas only a third of it has been isolated as water-insoluble methanol lignin.

It is of interest to note that the syringyl compound 1-(4-hydroxy-3, 5-dimethoxyphenyl)-1, 2-propanedione isolated by Kulka *et al.* (1944) from hardwoods has a methoxyl content of 27·7 per cent., whereas the corresponding guaiacyl compound has a methoxyl content of 16 0 per cent.

5. Conclusions

Extraction of the wood of *Eucalyptus regnans* F.v.M. with methanol at 150°C. takes into solution a large part of the lignin in a form which retains the reactions of lignin in the wood. There is, therefore, some justification for referring to this lignin as native lignin.

6. Acknowledgments

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Mineral Chlorination Studies

3. The Chlorination of Australian Beryl

By F. K. McTaggart, M.Sc.*

Summary

Optimum conditions for the decomposition by chlorine of beryl from Wodgina and Londonderry districts of Western Australia were determined and factors influencing the rate of reaction, such as fineness of grinding, catalysts, briquetting, and temperature, were investigated. Evidence was found for the presence of combined water in the beryls used. This was not eliminated at 1250°C. and prevented complete separation of the $AlCl_3$ and $BeCl_2$ produced when the latter was fractionally condensed in the range 200°–400°C. On resubliming the $BeCl_2$ and again condensing at 250°C. beryllium chloride free from aluminium was obtained. Other methods of separating the chlorides were investigated and it was found that among the organic solvents acetyl chloride is effective.

1. Introduction

During the last six or seven years the metal beryllium has assumed considerable importance both in the pure form and in the form of alloys of which beryllium–copper is perhaps the most widely used and best known. The demand for beryllium and its derivatives has increased steadily and although production has kept pace with requirements the processes in use for its production are not entirely satisfactory, as they involve some of the more difficult techniques known to industrial chemistry. Two main processes are in use, one depending on the electrolysis of the fused chloride and the other on the electrolysis of a fused complex fluoride. The former process may be carried out at relatively low temperatures (250°–800°C.) and low voltage (3–5 volts) but the beryllium is deposited in the form of flakes or powder and must be pressed and fused at 1300°C. under a suitable flux to yield ingots. The latter process yields the metal either in the form of an alloy or in the massive state but requires a working temperature of at least 1400°C. and a voltage of 70–80 volts. The present tendency overseas appears to favour the chloride electrolysis. Both processes involved, until recently, the breakdown of the ore by various means to yield intermediate compounds, for example, beryllium oxide, which was then chlorinated in the presence of carbon to yield anhydrous beryllium chloride.

Beryl, which is a silicate of beryllium and aluminium, is the only beryllium ore of general commercial importance. Notable deposits of beryl in pegmatite formations occur in certain parts of Australia, particularly in Western Australia at Wodgina, Londonderry, and elsewhere, and in the Boolcoomatta district of South Australia. These deposits have been worked to some extent but the beryl has been exported, and as yet no beryllium has been produced in this country. Because of the raw material potentially available it appears likely that Australia could satisfy a significant proportion of the present

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demand for beryllium if the ore were processed locally. The above considerations form the background against which the present investigation was undertaken.

Winters and Yntema (1) have described a process for the production of anhydrous beryllium chloride by direct chlorination of beryl, the other chlorides formed, namely aluminium chloride and silicon tetrachloride, being separated by passing them through a tube held at 375°C. Under these conditions the beryllium chloride which has a b.p. of 520°C. is said to condense in the tube in a pure form, while the chlorides of aluminium, sub. pt. 178°C., and silicon, b.p. 57·6°C., pass through. The present paper describes the application of this process to beryls of Australian origin and the investigation of the various factors which influence the rate of chlorination. Some data are appended concerning other means of separation.

The theoretical formula for beryl, which is a beryllium aluminium silicate, may be expressed thus: $3BeO, Al_2O_3, 6SiO_2$, i.e., 14 per cent. BeO, 19 per cent. Al_2O_3, and 67 per cent. SiO_2. In nature, however, these proportions are not adhered to strictly, the BeO varying from about 8 to 13 per cent. with corresponding variations in the percentages of the other constituents. Caesium, scandium, manganese, alkalis, and other elements may also be present in small quantities. The beryl crystal is one of the most complex silicates known, the Al, Be, and Si atoms being linked in a manner such as to bear no resemblance to oxide structure. As W. L. Bragg (2) says:—" The structure of beryl (aquamarine, emerald) may perhaps be considered as one of the most elegant of the many interesting coordinated structures displayed by silicates ". Fig. 1 is a reproduction, taken from this book, of the structure of beryl:

· a•9.21A ·
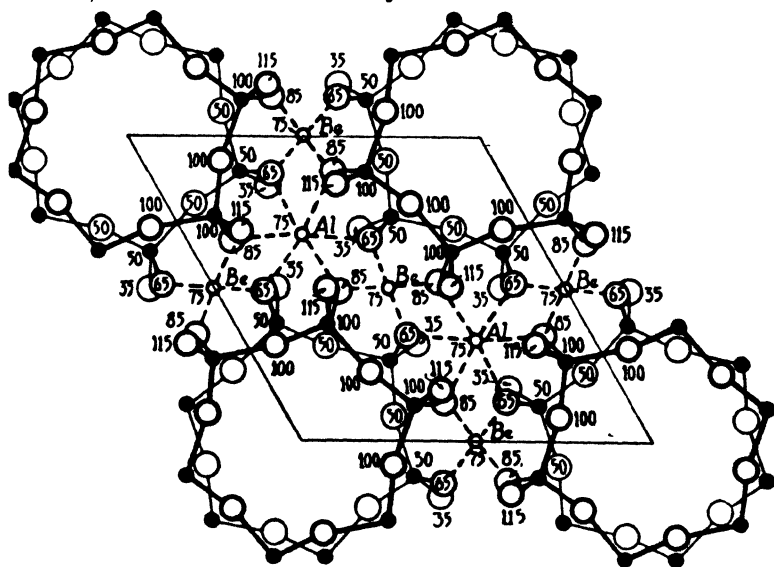
FIG. 1.—The structure of beryl, $Be_3Al_2Si_6O_{18}$. Note the Si_6O_{18} rings. Superimposed oxygen atoms are symmetrically displaced. The reflection planes are at heights 0, 50, 100. (Reproduced from Bragg, 1937.)

The composition (see next section) of the beryl used by us was, however, sufficiently close to the theoretical for use to be made of the latter in calculating the proportions of beryl and carbon for briquetting and the approximate heat requirements of the reaction.

The reaction is essentially as follows:—

$$3BeO + Al_2O_3 + 6SiO_2 + 18C + 18Cl_2 \rightarrow 3BeCl_2 + 2AlCl_3 + 6SiCl_4 + 18CO$$

	3BeO	Al₂O₃	6SiO₂	18C	18Cl₂	3BeCl₂	2AlCl₃	6SiCl₄	18CO
Mol. wts. ..	25	102	60	12	71	80	133·5	170	28
Mol. ratios by wt.	75	102	360	216	1278	240	267	1020	504
Wt. ratios ..		537		216	1278		1527		504
Heats of formation (△H) in kilocals	132?	389·5	200			112·5	166·8	149	26·4
Summation ..	396	389·5	1200			337·5	333·6	894	475·2

1985 K cals.   2040 K cals.

There are insufficient data available at the present time for the calculation of the quantity of heat given out, or absorbed, per mole at the reaction temperature, or even of the free energies at room temperature, but from past experience with chlorinations of this nature and a consideration of the data given above, it would appear that the reaction is likely to be slightly exothermic at the reaction temperature and on a large scale it must be considered likely that sufficient heat would be generated to make external heating unnecessary provided the charge was initially at a sufficiently high temperature. This procedure has, for example, been found to be completely successful in the industrial scale chlorination of rutile. In any case, the heat requirements of the reaction would be small.

There are few references in the literature to the chlorination of beryl although many workers have prepared anhydrous beryllium chloride by chlorination of the oxide. In addition to those of Winters and Yntema (1) the only other chlorinations carried out on beryl appear to be (a) that referred to in a patent held by Portnov and Seferovich (3) and (b) one described by Fink (1). However, in none of these accounts are any details of proportions, reaction temperatures, reaction rates, etc., given. Fink stated that coarsely-ground beryl, without carbon, chlorinated readily at a red heat, but we were not able to confirm this. Portnov and Seferovich claimed that the reaction may be catalysed by small amounts of alkali metal chlorides.

2. Raw Materials

(i) *Beryl*

The beryls used in this investigation were of the massive variety. That from Londonderry was pale green in colour and was obtained from a large pegmatite formation worked for microcline felspar. That from the tantalite locality at Wodgina was white in color and of the variety known as rosterite containing about 1 per cent. of

caesium oxide. Londonderry beryl, of the type used in the work described here, has been described by Le Mesurier (5) and typical analyses given by him are as follows:—

Color: Milky white to pale green.

	1st. sample per cent.	2nd. sample per cent.
SiO_2	63·40	63·80
Al_2O_3	21·28	17·26
Fe_2O_3	—	0·80
BeO	9·56	12·58
MgO	0·16	0·02
CaO	0·20	0·30
Li_2O	0·72	0·82
Na_2O	1·24	1·38
K_2O	0·16	0·20
Ign. loss	2·57	2·30
	99·29	99·46

The sample used contained 12·5 per cent. BeO and suffered the following losses on heating: to 120°C. 0·1 per cent., 120°–1000°C. 2·1 per cent., 1000°–1200°C. 0·2 per cent., making a total of 2·4 per cent. It was reserved mainly for the work on the separation of the chlorides because its composition approached more nearly that of the ideal beryl, 3BeO, Al_2O_3, $6SiO_2$, than the Wodgina ore. For this purpose it was crushed in a jaw crusher and ground in a roller mill to minus 10 mesh and finally ground in a porcelain ball mill to 98 per cent. minus 200 mesh.

Wodgina beryl has been described by Simpson (6) who gave the following analysis:—

SiO_2	66·42
Al_2O_3	17·97
Fe_2O_3	nil
BeO	11·20
FeO	nil
MnO	nil
MgO	nil
CaO	0·30
Li_2O	0·82
Na_2O	1·01
K_2O	trace
Cs_2O	0·72
Ign. loss	2·20
	100·64

Other samples were reported to contain MnO. That used in this work contained 0·4 per cent. MnO and showed a loss on ignition to 120°C. 2·3 per cent., 120°–800°C. 1·1 per cent., 800–1000°C. 1·0 per cent., 1000°–1200°C. nil, making a total loss of 4·4 per cent.

This ore was used in the determination of reaction rates and for the experiments showing the influence of particle size. It was ground in a similar manner to the Londonderry ore but was finally screened and these fractions collected: — 100 + 150 mesh; — 150 + 200 mesh; — 200 + 270 mesh; and —300 mesh.

(ii) *Coal*

The coal used in these experiments was a black bituminous type and was crushed and ground until it had the following screen analysis: 10 per cent. retained on 50 mesh screen, 41·5 per cent. retained on 100 mesh screen, 29·5 per cent. retained on 150 mesh screen, 12·0 per cent. retained on 200 mesh screen, 6·0 per cent passed by 200 mesh screen.

The coal contained 11·5 per cent. ash made up of 7·6 per cent. SiO_2, 0·5 per cent. Fe_2O_3, 2·6 per cent. of other R_2O_3, and 0·8 per cent. undetermined alkalis, etc.

(iii) *Charcoal*

High-grade activated charcoal as used for decolorizing purposes was employed for most of the experiments on reaction rate. The ash content of this material was 8·0 per cent.

(iv) *Tar*

A light fraction of re-distilled vertical still tar which had the following physical and chemical characteristics *:

Specific gravity	.. ..	1·06
Free carbon	.. ..	5 per cent. by weight
Water	.. ..	nil

Distillation range:—

0–170°C.	.. ..	nil
170°–230°C.	.. ..	8·6 per cent. (by volume)
230°–300°C.	. .	28 per cent. (by volume)
Total oils to 300°C.	..	36·6 per cent. (by volume)
Pitch content	.. .	63 per cent. (by weight)

This tar, which was thin and dark brown in color, was found to be ideally suited for briquetting purposes.

3. Experimental

(i) *Briquetting*

The reaction between beryl (having the theoretical formula 3BeO. Al_2O_3.6SiO_2), carbon, and chlorine has been shown above. From this it is seen that a briquetted mixture of beryl and carbon should contain 71 per cent. of beryl and 29 per cent. of carbon. However, this would only apply if the beryllium, aluminium, and silicon content of the mineral were attacked during chlorination to an equal degree. Initial experiments were made, using the above proportions, to determine whether equal attack did occur, and to this end a sample of briquettes

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* We are indebted to Messrs. James Forbes (Tar Distillers), Melbourne, for this analysis, which is stated in the conventional tar manufacturers' way. This specification is very close to what is known in Australia as No. 1 vertical tar and could be described as "No. 1 vertical tar plus 3 per cent. of light oils".

containing by analysis 72·8 per cent. total ash (beryl plus carbon ash) and 27·2 per cent. carbon was ashed and an analysis for SiO_2 made on this ash. A figure of 63·0 per cent. SiO_2 was obtained. Briquettes from the same batch were then chlorinated until 91·6 per cent. conversion of the total ash content had been achieved. The residue which contained 54 per cent. carbon was ashed and the SiO_2 content was then found to be 67·3 per cent. To show that this difference is not significant when related to the actual quantities involved, Table 1 has been prepared.

TABLE 1

137 *g. briquettes*	137 *g. briquettes*
chlorinated	ashed
18·2 g. residue	(100 g. beryl and carbon ash)
8·4 g. total ash	63 g. SiO_2

if 67·3 per cent. SiO_2 if 63 per cent. SiO_2

5·65 g. SiO_2 5·29 g. SiO_2

It shows the final discrepancy between the SiO_2 consumed theoretically (for uniform attack on the Al, Be, and Si content of the beryl) and that consumed actually, when 137 grams of briquettes were chlorinated. This discrepancy amounted to only 0·36 g. in 63 g. —less than 0·6 per cent. In other words the Al, Be, and Si in the beryl were, within close limits, attacked to an equal extent. Hence the theoretical proportions of beryl and carbon are correct, and the aim when making briquettes has always been to approximate closely the 71:29 ratio.

The method of briquetting has been fully described elsewhere (7), and was followed in this case both when using charcoal and coal. Too little carbon resulted in the briquettes crumbling badly during chlorination, while too much had no undesirable effect other than causing a waste of furnace space. The size of the briquette pieces used throughout the experimental work was minus ¼-in. mesh plus 1/32-in. mesh, in order to keep the conditions constant, but larger pieces up to ½-in. mesh or more were found to chlorinate satisfactorily, no diminution in reaction rate being noticeable.

The difference between briquettes made from coal and charcoal is shown later.

(ii) *The Laboratory Chlorination Furnace*

This is shown in Fig. 2. It consisted of a vertical silica tube furnace (A), wound with a heating element of 26-gauge platinum— 20 per cent. rhodium wire, and fitted with a chlorine inlet tube (B),

thermocouple tube (C), and side outlet (D) for the gaseous reaction products. This side outlet was wound with a nichrome heating element to maintain it at a temperature slightly above 500°C. (E) was an opening used for filling the furnace, this opening being closed during chlorination by the ground plug (F).
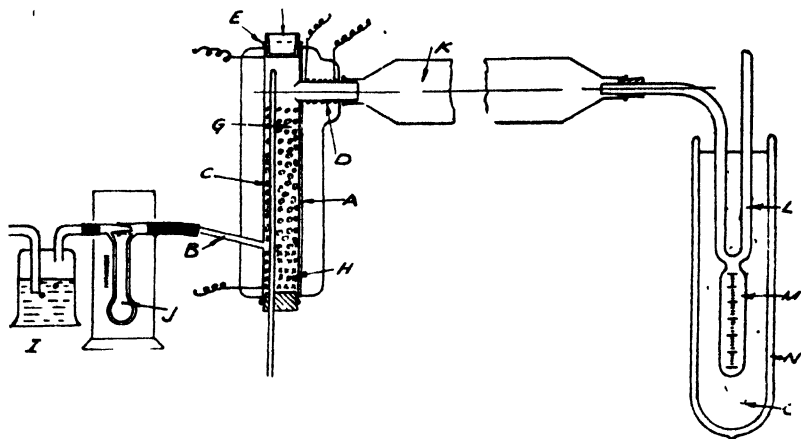
FIG. 2.—Laboratory chlorination furnace with apparatus for determination of reaction rates.

The charge (G) of briquetted beryl and carbon rested on a bed of silica chips (H), the latter ensuring that the whole of the charge was at a uniform temperature within the furnace. Chlorine was introduced into the furnace via the sulphuric acid bubbler (I) and the flowmeter (0–500 cubic centimetres per minute, orifice type) (J).

(iii) *Influence of Particle Size on Reaction Rate*

It has been shown that the aluminium, beryllium, and silicon in the beryl react during chlorination to an equal extent. Use was made of this fact in the determination of reaction rates. Owing to the difficulty of measuring the amounts of $BeCl_2$ and $AlCl_3$ produced during a given interval of time, these chlorides were condensed out completely and the $SiCl_4$ passed into a condenser. A measure of the volume of $SiCl_4$ collected in any given time, when other factors were constant (temperature, particle size, &c.) gave a measure of the reaction rate.

The apparatus used in addition to the chlorination furnace described above is shown on the right-hand side of Fig. 2. (K) is a pyrex glass tube 2 feet in length and $1\frac{1}{2}$ in. in diameter. It was attached to the side outlet (D) of the furnace and served as an air condenser for the trapping of the $BeCl_2$ and $AlCl_3$. At room temperatures the $SiCl_4$ passed through this tube and was condensed in the U tube condenser (L), which was fitted with a receiving section (M) graduated in cc. The latter, with both arms of the condenser, was immersed in absolute alcohol cooled to $-25°C$. by means of solid carbon dioxide, "dry ice." (N) was a vacuum flask containing the alcohol. A temperature above $-33\cdot6°C$., the boiling point of chlorine, was chosen to avoid condensation of the excess chlorine passing through the system.

Wodgina beryl screened as previously described to − 100 + 150, − 150 + 200, − 200 + 270, and minus 325 mesh was briquetted with B.D.H. decolourizing charcoal. The ash contents (beryl plus carbon ash) of these four batches of briquettes lay between the limits of 65 and 70 per cent. Charges of 100 g. − ¼-in + 1/32-in. mesh were used. The procedure was to place the charge in the furnace, close the top opening with the ground silica stopper, and attach the glass air condenser by means of a small amount of cement, consisting of equal parts of coarse asbestos fibre and alundum cement. The current was then turned on and the furnace and side outlet brought up to full working temperature. During this time a stream of dried nitrogen at 100-150 cc. per minute was passed through the furnace, and this was continued until all traces of moisture had been driven out of the system. The condenser for the $SiCl_4$ was then attached by means of a rubber stopper to the end of the air condenser and the vacuum flask containing the cooled alcohol placed in position. Chlorine was then passed into the furnace at a rate such that a slight excess appeared at the condenser outlet. This could be determined by analysis of the exit gases or more readily by calculation based on the amount of $SiCl_4$ condensing.

Table 2 shows the reaction rate, expressed as cc. of $SiCl_4$ evolved per fifteen minutes for the various sizes of beryl at temperatures of 800°, 1,000°, and 1,200°C.

TABLE 2

Briquette	Beryl Mesh	Reaction Rate cc of $SiCl_4$ per 15 mins		
		Temp 800° C	Temp. 1000° C	Temp. 1200° C
a	−100 +150	0·9	4·3?	4·2
b	−150 +200	2·5	4·3	4·8
c	−200 +270	3·5	4·9	5·1
d	−325	5·25	5·6	5·8

The influence of particle size is seen to be very marked at 800°C., but less so at 1000°C., and at 1200°C. It would appear necessary to grind the beryl at least to 90 per cent. minus 200 mesh. The reaction could then be carried out satisfactorily at 1000°C. Conversely, a temperature of 800°C. could be used with no great reduction in reaction rate, provided the beryl was ground to 100 per cent. minus 325 mesh. For large-scale work Table 2 allows a choice to be made and an optimum found between particle size and temperature.

(iv) *Influence of Temperature on Reaction Rate.*

In addition to the experimental work described above, reaction rates were determined at temperatures lower than 800°C., and the temperature at which reaction commenced was determined for each particle size. These results, together with those given in Table 2, are plotted in Fig. 3, the rate of reaction, given as volume in cc. of $SiCl_4$ per fifteen minutes, being plotted against temperature of reaction.

The shape of the curves is similar to that obtained for the reaction between rutile, carbon, and chlorine (7), and appears to be typical of heterogeneous reactions such as this. Reaction rate apparently depends on two factors:—

(a) The area of the solid surface which increases with degree of fineness, and the number of active surface spots which increases with temperature.

(b) The rate of diffusion of the chlorinating agent to, and the gaseous products away from, the solid surface which reaches a limiting value.
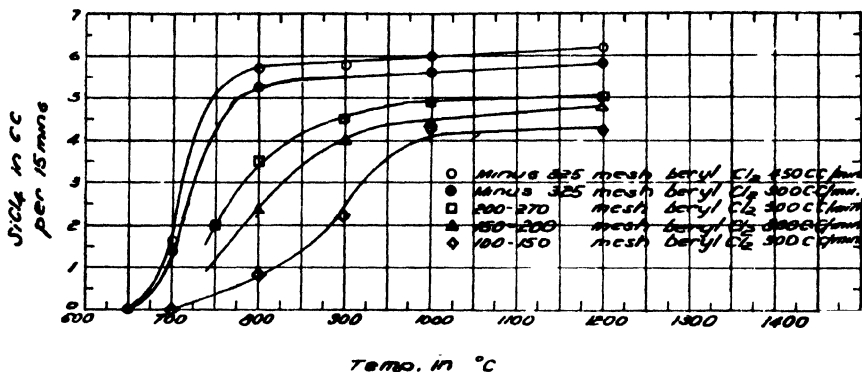
FIG. 3.—Influence of temperature and particle size on reaction rate

The use of a faster chlorine rate has the effect of promoting a more rapid removal of products from the active surface. This can be seen in the upper curve in Fig. 3, which was obtained when the chlorine rate was raised from 300 cc. per minute to 450 cc. per minute.

(v) *Influence of Charcoal and Coal on Briquettes*

In sections (iii) and (iv) all briquetting was done with B.D.H. decolourizing charcoal. The cost of a material such as this would obviously be prohibitive in large-scale work, and it was therefore necessary to examine the effect of coal as the source of carbon in the briquettes. Hence, samples were made from Wodonga beryl of minus
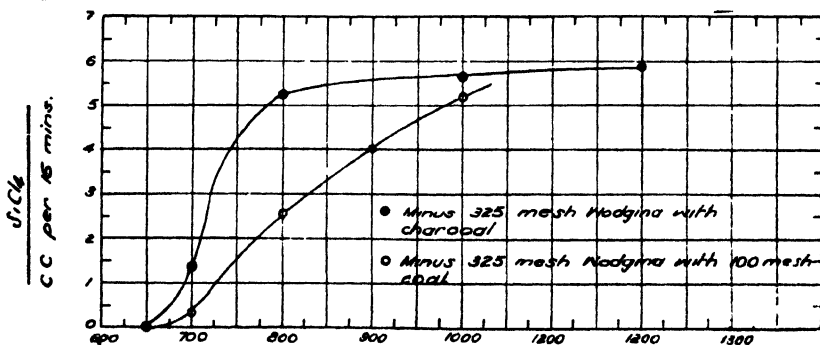
FIG. 4.—Reaction rates using coal and charcoal respectively.

325 mesh, 100 mesh coal, and tar, the briquetting being done in the standard manner. The porous masses produced were of a similar quality to the type previously used, had the same apparent porosity, and were of the same hardness. Reaction rates were determined at 1000°, 900°, 800°, and 700°C. The figures obtained are shown in graphical form in Fig. 4, together with a reproduction of the curve for Wodgina minus 325 mesh beryl plus charcoal.

There was little to choose between the coal and charcoal types at 1000°C., but at lower temperatures the former was unsatisfactory. It would appear that when using the coal-beryl briquettes a temperature of at least 900°C. is necessary for a reasonable reaction rate. Several batches of briquettes gave the same results.

(vi) *Effect of Addition of Alkali Metal Chloride on Reaction Rate and Difference between Wodgina and Londonderry Beryl*

Portnov and Seferovich (3) claimed that the reaction between beryl, carbon, and chlorine is catalysed by the addition of small, unstated quantities of alkali metal chlorides. Other workers have claimed similar results when chlorinating such minerals as rutile, ilmenite, and zircon. It was interesting, therefore, to apply the previously described technique for measuring reaction rates to this problem. At the outset it was realized that if these claims are correct the two types of beryl might be expected to show similar reaction rates since both have alkali contents of the same order. Therefore comparisons were first made between the two types. Both were ground to minus 325 mesh and briquetted with decolourizing charcoal and tar in the standard manner, the beryl-carbon ratio held within narrow limits. The variation of reaction rate with temperature for these samples is shown by curves (a) and (c) in Fig. 5.
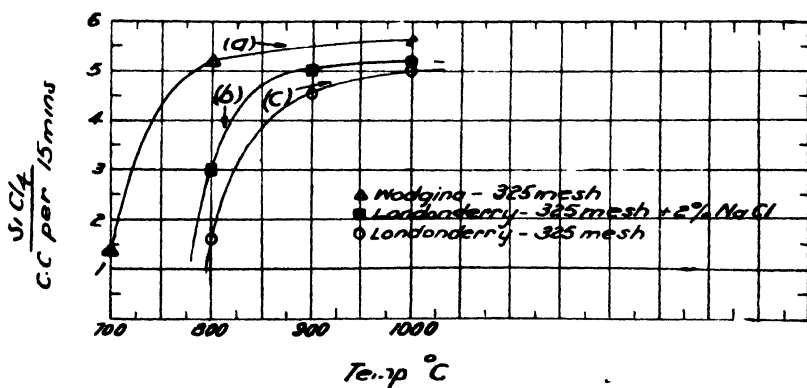
FIG. 5.—Reaction rates for different beryls and influence of alkali chloride.

It will be seen that there was a very marked difference between the two. The Wodgina beryl commenced to react at a temperature 100°C. below the Londonderry, and reacted faster at all temperatures up to 1000°C. No explanation can be given for this phenomenon, but it was evident that some other factor besides the alkali content was involved. The third curve shown (b) is for Londonderry beryl of minus 325 mesh plus 2 per cent. sodium chloride. To ensure good

That the vapours passed through the tube too quickly for temperature equilibrium to be established is disproved by No. 27, in which the chlorine rate was only 50 cc. per minute, and No. 21, in which it was 75 cc. per minute. No increased amount of chlorides was collected in these experiments over that collected in Nos. 36 and 39, so that the possibility of " sweeping out " of vapour by too rapid gas flow must be eliminated. In an attempt to achieve better separation a series of chlorinations was done with the collecting tube held at various temperatures between 200°C. and 400°C. The results are given in Table 5 and are graphed in Fig. 7.
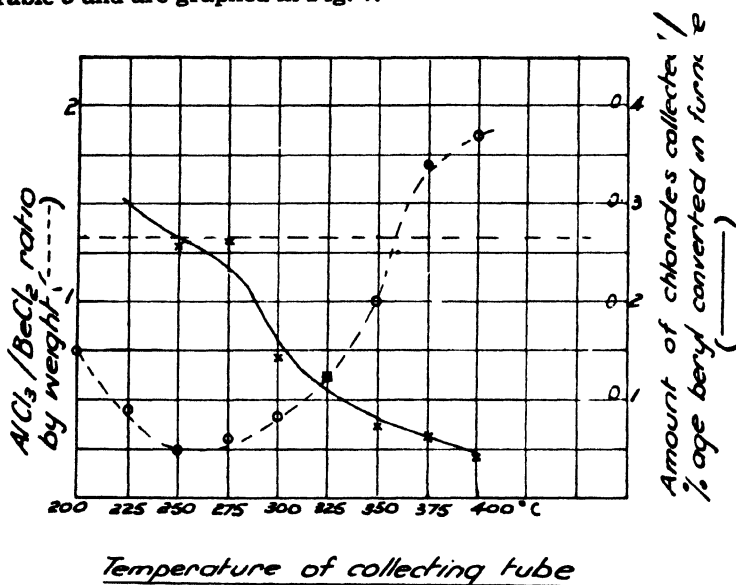
FIG. 7.—Influence of condenser temperature on selective deposition of AlCl$_3$ and BeCl$_2$.

It will be seen from Fig. 7 that there is a point of maximum enrichment of BeCl$_2$ at 250°C. The reason for this was not immediately evident, so further experiments were carried out for the purpose of elucidating the matter. Pure Al$_2$O$_3$ and BeO, both of which had been ignited to 1200°C., in the ratio of 17:10 by weight (corresponding approximately to the proportions of these oxides in beryl) were briquetted with charcoal in the standard manner and chlorinated at 900°-950°C. The AlCl$_3$ and BeCl$_2$ vapours formed were passed through the collecting tube which was held at 350°-360°C. It was found that pale yellow, almost white, crystals of BeCl$_2$ had formed in the tube and that the AlCl$_3$ had passed through. Thus the mixed oxides behaved very differently from the beryl. Two possible factors that might cause these anomalous results were recognized:—

    (a) The presence of SiCl$_4$ vapour in the case of the beryl might be the cause of formation of loose compounds, thus changing the boiling point of chlorides.

    (b) The presence of moisture during the chlorination in spite of the fact that the briquettes were heated to 800°C. prior to chlorination.

TABLE 5

Expt. No.	Temp. of Furnace °C.	Temp. Data		Residue Data			Chlorine Rate cc./min.	Time hr.	Chlorides Collected in Tube			Total Chlorides Obt. g.	Remarks
		Tube °C.	Tolerance °C.	Charge g.	Weight g.	Conversion %			Weight g.	AlCl₃/BeCl₂ Ratio by Weight	Weight (g.)/Percentage Beryl Converted		
45*	1000	350	±5	100	40	75	100	5	9·2	1	..	..	Briqs. not dried
46*	1000	300	"	100	..	..	100	5	..	0·4	..	..	Briqs. dried for 3 hr. at 1100° C.
56**	1100	300	"	100	25	80	200	4	..	0·06	..	..	Short collecting tube
42	980	200	±2·5	70	7	92	100	5	11·7	0·74	..	26	
41	950	225	"	70	8	90	100	5	15·4	0·37	..	32·4	
40	950	250	"	70	25	64	100	5	15·5	0·26	0·240	..	
17	950	250	"	70	14	80	100	4	9	0·25	..	21	
38	975	275	"	70	28	60	100	5	15·3	0·3	0·255	..	
33	975	300	"	70	10	86	100	5	13·6	0·4 } mean	0·143	28·6	
18	975	300	"	70	13	81	100	4	10	0·38	..	38?	
34	975	325	"	70	10	86	100	5½	10·7	0·6	0·125	33·2	
35	980	350	"	70	16·5	78	100	5	7·3	1 } mean	0·075	..	
25	980	350–360	"	70	37	47	100	5	3	1·3	..	18	Temp. a little higher than 350° C. at times
39	950	375	±2·5	70	27	62	100	5	3·5	2·5 } mean	0·060	13·6 }	Mean AlCl₃/BeCl₂ ratio (see Table 4 also) = 1·7:1
36	950	375	"	70	33	53	75	5	3·6	1·25	..		
27	950	400	"	70	13	80	75	5	3·5	1·8	0·043	30?	Lower Cl₂ rate in attempt to get more chloride

Mixed oxides (see text).

To investigate (a), a mixture of the oxides of aluminium, beryllium and silicon was made up, consisting of 19 per cent. Al_2O_3, 11 per cent. BeO, and 70 per cent. precipitated SiO_2—each of minus 300 mesh. This mixture was briquetted in the usual manner and chlorinated. Results are shown in Table 5. In Experiment No. 45 the collecting tube was held at 350°-360°C., while in No. 46 it was held at 290°-300C°. It will be seen that the figures for the $AlCl_3$: $BeCl_2$ ratio agree closely with those obtained using beryl (e.g., Experiments 33 and 35). At first sight this appeared to confirm the view that the $SiCl_4$ was the disturbing factor, but it was considered desirable to investigate (b) thoroughly before coming to a definite conclusion.

The occurrence of a minimum point on the curve (Fig. 6), which indicated maximum enrichment of $BeCl_2$ at 250°C., may be explained by assuming that a practically constant fraction of the $AlCl_3$ produced in every chlorination was hydrolysed, and that this material collected in the tube, together with the anhydrous $BeCl_2$. There might also have been a small amount of $BeCl_2$ hydrolysed each time—but this would be less in amount than the $AlCl_3$, see below. Then at temperatures lower than 250°C. larger quantities of $AlCl_3$ would be obtained owing to condensation of unhydrolysed $AlCl_3$, which would have the effect of increasing the $AlCl_3$: $BeCl_2$ ratio, while at temperatures higher than 250°C. smaller and smaller quantities of $BeCl_2$ would be retained; the hydrolysed $AlCl_3$ would still be present and the net effect again would be to increase the $AlCl_3$: $BeCl_2$ ratio. That the $AlCl_3$ would be preferentially hydrolysed in this manner is in agreement with thermo-dynamical data for the two chlorides, and that the total $BeCl_2$ collected falls off gradually between 250°C. and 400°C. in agreement with the above reasoning is shown by the downward slope of the collection curve (also Fig. 6). At high temperatures practically nothing was collected in the tube, the material present being in the form of a brown sludge on the bottom of the tube. It appeared certain that this material was all in the hydrolysed form.

If this hydrolysis was taking place the moisture could come only from the charge because the chlorine was thoroughly dried through sulphuric acid and calcium chloride tubes, and furnace and collecting tubes were always dried for several hours at their respective running temperatures before chlorination was commenced. Firstly therefore, the mixed oxide briquettes described above were heated for several hours at 1100°C. and then chlorinated. The results (Experiment 56, Table 5) showed a marked improvement on previous chlorinations, the $AlCl_3$: $BeCl_2$ being 0·06 : 1, compared to the previous best 0·25 : 1 As this demonstrated conclusively that moisture in the charge could cause lack of separation, attention was turned to beryl briquettes. In a series of experiments summarized in Table 6 beryl briquettes were dried at 1200°C. for 4–5 hours immediately prior to being chlorinated.

In Experiments Nos. 57, 59 and 60, briquettes were dried in the furnace itself in an atmosphere of nitrogen, and were then chlorinated at the same temperature—i.e., 1200°C. With the collecting tube at 370°C. little or nothing was retained while at 250°C. the ratio of $AlCl_3$ to $BeCl_2$ was 0·2 : 1, but analysis showed (column 14 and 15, Table 6) that some $AlCl_3$ was condensing. At 330°C. the ratio was 0·1 : 1 which was considered more satisfactory. However, when similar chlorinations were carried out at 1000°C.—a

TABLE 6

Expt. No.	Charge Wt. (g.)	Charge Ash (%)	History of Charge	Furnace Temp (°C.)	Cl. Rate—cc./min.	Time (hr.)	Residue Wt. (g.)	Residue Ash (%)	SiCl₄ Cond. (g.)	Reaction (%)	Tube Temp. (°C.)	Loose Crystals Wt. (g.)	Loose Crystals AlCl₃/BeCl₂ Wt. Ratio	Remainder Wt. (g.)	Remainder AlCl₃/BeCl₂ Wt. Ratio	Overall Ratio AlCl₃/BeCl₂ in Tube	Percentage Total BeCl₂ Produced Retained in Tube	Percentage Total AlCl₃ Produced Retained in Tube	Glass Condenser Wt. of A.C.₃ (g.)	Glass Condenser Wt. of BeCl₂ (g.)	Remarks
60	100	67	Dried 4 hr. in furnace at 1200° C.	1200	220	3¾	20	..	81	82	250	8	0·035	13·1	0·37	0·2	80	15	17·2	4·6	Temp. 250° too low
59	"	"	"	"	"	4½	22	80	78	79	330	5	0·04	6	0·17	0·1	55	5	..	..	Optimum temp.?
57	"	"	"	"	200	3½	..	..	75	7·?	370	Practically nil		nil	collected	collected	..	..	17·7	16·7	Temp. 370° C. too high
61	"	"	"	1000	150	4	38	..	63	63	300	5	0·038	8·6	0·43	0·23	78	13·7	18·5	4·1	Temp. 350° too high
75	"	"	"	"	100	4½	39	..	57	60	330	5	0·058	9·2	0·62	0·33	70	16	17·5	4·5	
77	96	"	"	"	150	5	26	..	71	75	350	4	0·125	5	3·3	0·6	26	15	20·7	8·6	
78	"	65	Wodgina beryl, dried at 800° C.	"	100	4½	24	75	63	65	330	..	..	..	..	0·8	..	..	..	..	Cf. Expts. 34 and 35, Table 5
79	75	"	Wodgina beryl dried 5 hr. at 1200° C.	"	150	5	24	70	52	70	"	..	..	..	..	0·25	60	14	9·2	4·8	Cf. 61 and 75 above

temperature that must be regarded as a maximum for this type of reaction, owing to the rapid attack on silica, &c. by chlorine at high temperatures—results were disappointing. In Experiments 61 and 75 the charges were heated to 1200°C. for 4–5 hours in an electric muffle prior to chlorination, but the separation was little better than in previous cases cited in Table 5. Increasing the temperature of the collecting tube to 350°C. (Experiment 77) had the effect of reducing the amount of $BeCl_2$ retained, so that it appeared that 300°–330°C. was the optimum temperature for collection. Wodgina beryl briquettes treated and chlorinated under the same conditions (Experiment 79) gave almost identical results. It was found in these experiments that a quantity of the $BeCl_2$ was always condensed out in large, well-formed crystals on the upper part of the collecting tube and could be mechanically separated (see column 11, Table 6, Experiments 59, 61 and 75). These crystals seldom contained more than 4 per cent. of $AlCl_3$ but did not amount to more than 30–40 per cent. of the total $BeCl_2$ formed.

The use of baffles designed to bring about a more complete separation of these crystals was tried but was not successful. The bulk of the $AlCl_3$ in the tube was always in the brownish sludge on the bottom. It appeared to be partially hydrolysed and was a liquid above 250°C. No reason can be given at the present time for the increased separation found when chlorinating at 1200°C., but this temperature must be regarded as impracticable for general use.

The results obtained from all these experiments pointed clearly to the presence of moisture in the charge which, during chlorination, combined with part of the $AlCl_3$ to form a compound involatile up to 400°C. The question as to the source of this moisture remained unanswered. Kleeman (10) has shown that beryl from Boolcoomatta, South Australia, contains approximately 1·74 per cent. water given off between 120° and 800°C. He suggested that most of the water is given off at about 700°–800°C. but apparently did not confirm this. It appeared that four-hydroxyl groups replace one silicon-oxygen group in this type of beryl. We found that Londonderry beryl lost 1·95 per cent. water between 120° and 800°C., a further 0·15 per cent. between 800° and 1000°C., and suffered a further loss of weight of 0·2 per cent. between 1000° and 1200°C., which, on the evidence of the chlorination results, was still more water. Wodgina beryl lost 1·05 per cent. between 120° and 800°C., 1·05 per cent. between 800° and 1000°C., and practically nil between 1000° and 1200°C. Neither beryl can be heated above 1200°C. without sintering and fusing. The water remaining above 800°C. in either case would be sufficient to prevent complete separation as obtained by Winters and Yntema, and in view of our results it is a matter of doubt whether all the combined water is driven off even at 1200°C. Beryl from other sources may not contain hydroxy groups such as must be present in the types used by us, and with such "anhydrous" ores it should be possible to achieve separation readily. It must be concluded, however, that this is not possible with Londonderry or Wodgina beryl.

4. Resublimation of Mixed Chlorides

An attempt was made to effect a separation of the aluminium and beryllium chlorides by condensing them in a glass tube at room temperature and resubliming at temperatures of 360°–420°C. The

apparatus used is shown in Fig. 8. A 10-in. length of 2-in. diameter pyrex glass tubing (A) carrying a ground glass joint (B) was sealed to the side outlet of the chlorination furnace (C), and surrounded over a 6-in. length immediately adjacent to the furnace outlet, by a sliding heating element (D) the temperature of which was maintained at 430°C. The chlorides of aluminium and beryllium condensed in the region shown while the silicon tetrachloride passed through the tube and was condensed in the previously described condenser. Chlorination
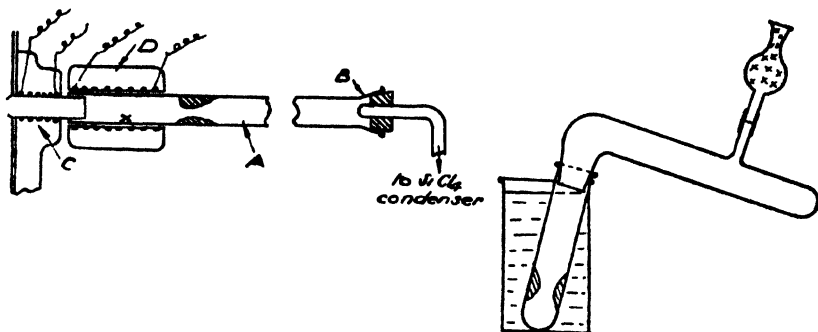
FIG. 8.—Differential sublimation apparatus.

of the charge was carried out in the usual manner with full precautions for drying, &c. At the conclusion of chlorination the whole system was swept out with nitrogen for ½ hour. The pyrex tube was then removed, quickly stoppered at each end with calcium chloride tubes, and sealed off at the position (X) in the blowpipe flame. A right-angled condenser tube (E) was fitted and the arm containing the chloride mixture was immersed in a salt bath (F) consisting of equal parts of sodium nitrate and sodium nitrite. The temperature of the bath was raised slowly to the desired value and left for ½ hour. The chlorides from each section were then dissolved out with dilute hydrochloric acid and analysed. The results are summarized in Table 7.

TABLE 7

Expt. No.	Total Wt. of AlCl₃ and BeCl₂ Approx.	Temp. of Sublimation	Residual Chloride		Sublimed Chloride		Remarks
			Total	BeCl₂/ AlCl₃	Total	BeCl₂/ AlCl₃	
	g.	°C.	g.				
1	30	420	13·9	2·1 : 1	15·8	1 : 3·6	BeCl₂ volatilized
4	16	360	9	2·5 : 1	7	1 : 13	

It will be seen that the separation resulting from resublimation was in all cases very poor and again evidence was obtained for the presence of hydrolysed aluminium chloride. Thus in No. 1, Table 7, heating the mixture to 420°C. did not drive out a significantly greater quantity of this material than heating to 360°C. (No. 4). However, a quantity of beryllium chloride was lost at the higher temperature.

5. Resublimation of Beryllium Chloride

Since the beryllium chloride condensed in the range 225°–275°C. was contaminated with aluminium chloride in a form that appeared to be substantially involatile up to 400°C. it was anticipated that resublimation would yield a purer product. Furthermore, to avoid traces of $AlCl_3$ condensing with the resublimed $BeCl_2$ it seemed desirable to condense the latter again in a tube maintained at 250°C. Such experiments were carried out with success. Beryllium chloride containing 20 per cent. $AlCl_3$ was prepared in the manner and with the apparatus already described, with the condenser temperature at 250°C. (Fig. 6). When sufficient had been collected, the passage of chlorine was stopped, the glass bulb was removed from the end of the condenser and a second silica tube, similar to the first was attached. This was maintained at 250°C. and was fitted with a glass bulb at the end. The temperature of the first tube was then slowly raised to 420°C. during which a stream of nitrogen was passed through the system. On completion of the experiment the second tube was found to contain a mass of pure white, needle-like crystals of $BeCl_2$, in which no trace of aluminium could be detected using 8-hydroxy-quinoline reagent. In the first tube 2–3 grams of chlorides was usually obtained, these being in a semi-liquid or liquid form, and containing 40–60 per cent. $AlCl_3$. In the bulb at the end of the system was found a similar quantity of dry chlorides containing 60–70 per cent. $AlCl_3$.

The yield of $BeCl_2$ was approximately 40 per cent. based on the amount of beryl decomposed in the furnace. This rather low figure was probably due to the nature of the condensing tubes, a loss being sustained during each condensation; and it is probable that the use of a more efficient type of condenser would improve the yield. Data from two typical experiments are given below:—

TABLE 8

No.	Beryl Consumed	After Resublimation						Yield $BeCl_2$	First Bulb	
		First Condenser		Second Condenser	Second Bulb					
		Wt.	$\frac{AlCl_3}{BeCl_2}$	$BeCl_2$ Wt.	Wt.	$\frac{AlCl_3}{BeCl_2}$			Wt.	$\frac{AlCl_3}{BeCl_2}$
	g.	g.		g.	g.		%	g.	
3	40	2·2	0·8	5·0	3·0	2·0	42	19	3·75
4	45	2·0	1·2	5·4	3·2	2·0	39	20	4·0

6. Separation by Means of Solvents

Wurster (11) suggested the use of certain chlorides or oxychlorides of sulphur, carbon, phosphorus, and boron for the separation of the anhydrous chlorides of aluminium and beryllium. In each case the aluminium chloride was claimed to be readily soluble and the beryllium chloride practically insoluble. No figures were given for the solubilities. Compounds of practical interest that occur in this class include $SOCl_2$ b.pt. 78°C., $COCl_2$ b.pt. 8·2°C., and $POCl_3$ b.pt. 107°C. Attention was first turned to phosphorus oxychloride because it has

a lower volatility and is less poisonous than the others. The solubilities of the two chlorides were determined in this solvent at 25°C. and were found to be—

$AlCl_3$ 14·4 per cent., and $BeCl_2$ 20·6 per cent.,

a result which is in the opposite sense to that expected and which means that separation by this means is impracticable. Aluminium chloride readily forms an addition compound with phosphorus oxychloride especially if mixtures of the two are warmed. The whole mass may become solid and much larger quantities of the solvent than those given by Wurster were found to be necessary for complete solution of the aluminium chloride.

There is other evidence (Booth and Torrey, 12) that beryllium chloride is only slightly soluble in carbonyl chloride. However, the use of such a toxic compound must be regarded with disfavour for large scale work. It was thought that acetyl chloride, CH_3COCl b.pt. 52°C., being a similar nature might prove a useful substitute, and the solubilities of the two chlorides in it were determined.

TABLE 9

| Temperature | Solubility | |
	BeCl₂	AlCl₃
°C.	%	
0	3·8	" miscible "
25	9·0	"

Aluminium chloride may be dissolved in acetyl chloride in any proportion, the solution gradually becoming more viscous. For example, 8 g. of anhydrous chloride in 15 g. of acetyl chloride yields a syrup of the consistency of treacle. As the above figures appeared promising a series of experiments was carried out using mixtures of the chlorides made synthetically and from the chlorination of beryl. The results are summarized in Table 10.

TABLE 10

| Expt No | Approx Wt of AlCl₃ | Approx. Wt of BeCl₂ | Volume of CH₃COCl | Temp. | Recovered BeCl₂ | | | Remarks |
					Wt	BeCl₂	AlCl₃	
	g.	g.	cc.	°C.	g.	%	%	
1	4	5	20	0	3·8 ⎫	73	3·4	Washed BeCl₂
2	4	5	20	0	3·5 ⎭			on filtrate with 5 cc. CH₃COCl at 0° C.
3	6·4	5·8	25	0	4·3	74	5	
4	8·1	7·4	30	0	4·8	65	1·3	Washed with 10 cc. CH₃COCl at 0° C.
5	10 g. mixed from beryl chlorination		30	0	3	..	4·5	BeCl₂ not washed on filter

It will be seen that a considerable degree of separation may be achieved by the use of acetyl chloride, which may be recoverd readily by distillation. Ferric chloride is readily soluble in acetyl chloride and is therefore removed with the aluminium.

7. Conclusion

An attempt was made to separate the chlorides of aluminium and beryllium by fractional condensation following direct chlorination of two beryls of Australian origin. It is believed that the presence of (OH) groups in the crystal lattice of the beryls was responsible for the failure to effect separation. Attempts to separate the aluminium chloride from the beryllium chloride by first collecting both chlorides and then subliming off the former by careful control of temperature were not successful. However, beryllium chloride collected at 250°C. and containing 10-20 per cent. aluminium chloride was obtained free from aluminium by resubliming and again condensing the beryllium chloride in a tube maintained at 250°C.

Acetyl chloride was found to be effective in separating the two chlorides but phosphorus oxychloride was not suitable. The solubilities of aluminium and beryllium chlorides in phosphorus oxychloride and acetyl chloride were determined.

8. Acknowledgments

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Studies in Cement-Aggregate Reaction

VII.—The Effect of Storage Conditions on Expansion and Tensile Strength Changes of Mortar

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Summary

Mortars containing high-alkali cement and reactive aggregate were stored under various conditions. Although expansion was affected by the environment (negatively in dry air; positively in other conditions), cracks were produced and significant reductions in tensile strength occurred in all mortars.

1. Introduction

It was shown by Stanton[†] (1940) that small mortar test pieces containing reactive aggregate and high-alkali cement expanded abnormally in a moist but not in a dry atmosphere. This suggests that different storage conditions, by affecting the amount of uncombined water in mortar, modify the expansion of reacted aggregate particles. Although similar mortars stored in water did not expand abnormally, more recent work by Stanton[**] (1943) has shown that storage in 1 and 2 per cent. sodium hydroxide solutions caused significant expansions. An attempt is made in this paper to show how storage conditions affect the expansion and the tensile strength of mortar.

2. Materials

TABLE 1.—CHEMICAL COMPOSITION OF CEMENT

Oxide	Percentage
SiO_2 ..	22·14
Fe_2O_3	2·38
Al_2O_3	4·94
CaO ..	64·12
MgO	1·08
SO_3 ..	1·84
Na_2O	0·45
K_2O ..	0·81
Loss on ignition	1·88

The reactive aggregate was a mixture consisting of five parts by weight of the reactive component plus 95 parts of the non-reactive component. The reactive component was an opaline rock (V.17) from Butcher's Ridge, East Gippsland, Victoria, crushed and sieved to — 18 + 52 B.S.S. mesh, and the non-reactive component was a quartz sand from either Leighton Buzzard, England, or Paringa, South Australia. These non-reactive sands were used in control mortars. Unless otherwise specified Leighton Buzzard sand was the non-reactive component used. The water : cement : aggregate ratio of all mortars was 0·5 : 1 : 2 by weight at the time of mixing.

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[*] An officer of the Division of Industrial Chemistry.
[†] Stanton, T. E. (1940).—*Proc. Amer. Soc. Civ. Engrs.* 66: 1781.
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3. Experimental

(i) *Mortar Bar Expansions*

Mortar bars, made from cement and reactive aggregate, were stored at room temperature under the following conditions: (*a*) in dry air*, (*b*) in moist air, (*c*) in 1 per cent. sodium hydroxide solution, and (*d*) in water.

After storage for 41 days some test bars from each were changed over to each of the other three storage conditions, and kept in these for the remainder of the test period. In this way the effects of pairs of storage conditions were observed. Some test bars were kept for the full test period in their initial environments. Mortar blocks of similar composition, stored under the same conditions as the test bars, were broken periodically and examined by means of a binocular microscope at 30 diameters magnification.

The expansions of these bars stored under different conditions are shown in Table 2. During the first 41 days storage the water vapour pressure in dry air storage may have exceeded 1 mm. Hg, since the partially spent calcium chloride was not replaced by the fresh desiccant frequently enough. As the results are not of exact quantitative significance this is unimportant.

Conclusions from mortar expansions are:—

1. Mortars did not expand significantly when stored in dry air. When stored in water they expanded very slowly but when stored in moist air or 1 per cent. sodium hydroxide solution they expanded rapidly.

2. Unexpanded motars taken from dry air and placed either in moist air, 1 per cent. sodium hydroxide solution, or in water, expanded very rapidly for a short time, after which their expansion rates decreased. These initial expansion rates were so high that it seemed probable that opal had undergone considerable attack by alkalis during storage in dry air and that when water was made available it was absorbed rapidly.

3. Expanded test bars, stored initially in moist air, expanded rapidly for a short time on being changed to water or 1 per cent. sodium hydroxide solution, after which their expansion rates decreased. This is similar to the previous result (2) and indicates that the reaction product can absorb large amounts of water in a short time.

4. Mortar which had expanded in 1 per cent. sodium hydroxide solution contracted slightly when changed to moist air but later expanded rapidly. When changed to water similar bars continued expanding very slowly. This suggests that the alkali (or sodium hydroxide) content of the surrounding solution affects the expansion rate of mortar.

5. Bars initially stored in water expanded excessively when changed to 1 per cent. sodium hydroxide solution. This suggests that either the increased concentration of alkalis in the storage solution

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* Dry air is a term used here in contrast with moist air. It refers to storage in an atmosphere above calcium chloride ($CaCl_2 - CaCl_2.H_2O$) and having a water vapour pressure less than 1 mm. Hg at 25°C.

TABLE 2.—PERCENTAGE EXPANSIONS OF TEST BARS

Time after Fabrication (Days)	Dry Air				Moist Air				1 per cent. Sodium Hydroxide Solution				Water			
	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
7	−0·019	−0·013	−0·014	−0·014	+0·005	+0·013	+0·007	±0·000	+0·011	+0·019	+0·009	+0·017	+0·017	+0·023	+0·013	+0·012
12	−0·037	−0·027	−0·018	−0·026	0·007	0·020	0·027	+0·015	0·032	0·055	0·047	0·051	0·017	0·027	0·016	0·014
18	−0·042	−0·027	−0·020	−0·030	0·165	0·177	0·189	0·150	0·144	0·188	0·177	0·186	0·024	0·029	0·020	0·023
27	−0·029	+0·031	+0·035	−0·018	0·497	0·498	0·521	0·438	0·301	0·349	0·344	0·348	0·067	0·042	0·046	0·058
34	−0·015	−0·051	0·064	−0·017	0·631	0·658	0·673	0·592	0·375	0·437	0·431	0·424	0·088	0·053	0·060	0·072
41*	−0·004	−0·061	0·067	−0·005	0·743	0·769	0·784	0·692	0·446	0·502	0·500	0·501	0·099	0·061	0·065	0·086
Changed to—	Dry Air	Moist Air	1 per cent. Sodium Hydroxide Soln.	Water	Dry Air	Moist Air	1 per cent. Sodium Hydroxide Soln.	Water	Dry Air	Moist Air	1 per cent. Sodium Hydroxide Soln.	Water	Dry Air	Moist Air	1 per cent. Sodium Hydroxide Soln.	Water
46	+0·012	+0·225	+0·182	+0·097	+0·773	+0·859	+0·994	+0·906	+0·417	+0·491	+0·555	+0·517	+0·063	+0·051	+0·075	+0·090
48	0·002	0·260	0·179	0·099	0·760	0·851	0·993	0·904	0·404	0·489	0·565	0·517	0·049	0·049	0·073	0·090
50	−0·004	0·286	0·186	0·099	0·751	0·855	0·992	0·903	0·399	0·482	0·582	0·516	0·047	0·044	0·074	0·090
55	−0·011	0·387	0·242	0·115	0·736	0·852	0·996	0·907	0·387	0·485	0·622	0·539	0·035	0·045	0·085	0·095
63	−0·027	0·511	0·293	0·122	0·714	0·862	0·994	0·902	0·365	0·546	0·679	0·556	0·020	0·042	0·118	0·094
70	−0·033	0·570	0·350	0·136	0·707	0·863	1·000	0·906	0·355	0·715	0·732	0·562	0·010	0·044	0·152	0·102
84	−0·044	0·709	0·409	0·142	0·682	0·924	0·999	0·906	0·332	0·965	0·797	0·579	−0·006	0·039	0·289	0·101
98	−0·050	0·809	0·473	0·146	0·669	0·983	1·005	0·908	0·320	1·117	0·888	0·603	−0·018	0·047	0·486	0·107
112	−0·062	0·878	0·521	0·148	0·659	1·046	1·007	0·910	0·308	1·173	0·950	0·600	−0·039	0·057	0·671	0·107
140	−0·064	0·988	0·592	0·147	0·649	1·198	1·009	0·910	0·295	1·322	1·048	0·605	−0·054	0·078	0·980	0·108
196	−0·071	1·023	0·667	0·155	0·641	1·362	1·025	0·914	0·285	1·469	1·156	0·603	−0·057	0·093	1·370	0·110
252	−0·072	1·206	0·701	0·154	0·645	1·409	1·048	0·917	0·289	1·583	1·225	0·606	−0·055	0·122	1·576	0·111
308	−0·113	1·206	0·712	0·167	0·644	1·538	1·091	0·917	0·288	1·692	1·255	0·603	−0·059	0·122	1·703	0·113
364	−0·116	1·205	0·743	0·157	0·640	1·546	1·124	0·918	0·284	1·692	1·287	0·606	−0·058	0·122	1·854	0·116

* Test bars changed to different storage conditions 41 days after fabrication.

prevents the continued diffusion of alkalis out of the mortar or, more probably, that the opal is being attacked by sodium hydroxide which diffuses from the storage solution into the mortar. When similar mortars were changed from water to moist air they contracted slightly and then, after a period of comparative stability, expanded slowly. This is probably due to the mortar containing little alkali and much uncombined water.

6. Mortar bars stored initially either in moist air, in 1 per cent. sodium hydroxide solution or in water, showed reduced expansion rates when changed to dry air. Their eventual contractions indicate that the availability of uncombined water affects the expansion of re-acting opal or the reaction product.

Observations on these mortars are summarized (a) after the first 41 days storage in each environment, and (b) after subsequent storage.

(a) Storage for first 41 days

(1) *In dry air.*—A few small spots developed on the surface of the mortar. When freshly broken the cement appeared dry. Opal particles were slightly softened but not wet. Numerous short very fine cracks in the mortar were associated with opal particles.

(2) *In moist air.*—Numerous wet spots developed on the mortar surfaces. Cement in freshly broken mortar appeared slightly moist. Changes observed in opal particles during reaction were: (a) soften-ing, (b) a tendency to form a jelly-like product, (c) some were finally converted to viscous liquid. The numerous cracks through the mortar were always associated with reacting opal particles and were filled only for part of their length with the reaction product.

(3) *In sodium hydroxide solution* (1 *per cent.*).—The mortar surfaces were coated thickly with gelatinous material. In freshly broken specimens the cement appeared very wet especially near original external surfaces. Opal particles throughout the mortar had been attacked and in many cases had been converted to viscous liquid. The mortar was cracked extensively and the cracks were com-pletely filled with reaction product.

(4) *In water.*—Only a few spots of white gelatinous material developed on the surfaces of the mortar. Cement in freshly broken specimens appeared very wet. Opal particles had reacted and softened and in some cases had been converted to viscous liquid. The mortar was traversed by numerous fine cracks which were associated with reacted particles. The cracks were completely filled with the reaction product.

(b) Storage from 41 days to 364 days

(1) Mortars changed from dry air to moist air, to 1 per cent. sodium hydroxide solution, and to water, cracked extensively and their reactive particles underwent considerable change. In moist air storage the reaction product appeared to remain in the gel state and did not fill the cracks completely. In 1 per cent. sodium hydroxide solution and in water storage the cracks were completely filled with reaction product which appeared to have been converted to viscous liquid.

(2) All mortars, when changed to dry air, dried out slowly. The reaction product in cracks and in spaces occupied originally by opal particles, appeared to shrink considerably.

(3) Cracks in mortars changed from moist air to 1 per cent. sodium hydroxide solution or to water became completely filled with reaction product.

(4) Mortars changed from water to 1 per cent. sodium hydroxide solution and *vice versa* showed very little change. The reactive particles in mortars originally stored in water appeared to be more completely attacked and softer after storage in 1 per cent. sodium hydroxide solution.

(5) Mortars originally stored in 1 per cent. sodium hydroxide solution or in water showed no change after storage in moist air.

(6) All mortars retained in their original environments showed no apparent changes.

(ii) *Losses from Mortars by Heating at* 110°C.

Some mortar blocks were made with non-reactive aggregate and some with reactive aggregate. These mortars, stored under the various conditions for periods up to 114 days, were heated in an electric oven for one hour at 110°C.

TABLE 3.—THE PERCENTAGE LOSSES FROM MORTARS AT 110°C

Aggregate	Time after Fabrication (Days)	Dry Air	Moist Air	Sodium Hydroxide Soln. (1 per cent.)	Water
Non-reactive .	1	11·0	11·0	10·7	10·8
	8	5·9	8·6	9·5	9·3
	21	4·0	8·0	9·5	9·4
	42	3·1	8·0	9·2	9·1
	77	2·1	7·5	8·9	9·0
	114	1·5	7·5	8·6	8·8
Reactive ..	1	11·0	11·6	11·3	11·4
	8	6·6	8·2	9·7	9·5
	21	4·1	8·2	10·3	9·3
	42	3·4	8·3	10·7	9·6
	77	2·4	8·0	10·7	8·9
	114	1·8	8·2	10·8	9·4

The results, which are shown in Table 3, indicate the relative amounts of uncombined water in each mortar. It is recognized that these losses are of only qualitative significance since some combined hydrate water is lost at this temperature and carbon dioxide is absorbed by the cement during heating. As all the losses at any given time should have an approximately constant error, the following conclusions concerning the amounts of uncombined water in mortar stored under the various conditions seem reasonable:—

(1) The amount of uncombined water in mortar stored in dry air was much lower than that in mortar stored under other conditions.

(2) The amount of uncombined water in mortar stored in moist air was lower than that in mortar stored either in 1 per cent. sodium hydroxide solution or in water. The differences were comparatively small.

(3) The somewhat high losses from reactive aggregate mortar when stored in 1 per cent. sodium hydroxide solution were probably due to the large quantity of water in the reaction product.

(iii) *The Behaviour of Alkalis in Mortar Stored in Water and 1 per cent. Sodium Hydroxide Solution*

Blocks of mortar, each weighing approximately 110 g., and of similar composition to those used in the experiments previously described, were stored in 250 ml. of each medium at room temperature. Some results indicating the behaviour of alkalis are shown in Table 4.

TABLE 4.—THE AMOUNTS OF SODIUM AND POTASSIUM HYDROXIDES FOUND IN SOLUTIONS IN WHICH MORTARS WERE STORED

Pre-treatment	Time in Storage Solutions (Days)	In Water		In 1 per cent. Sodium Hydroxide Solution	
		Non-reactive Aggregate	Reactive Aggregate	Non-reactive Aggregate	Reactive Aggregate
Mortars placed in storage solutions 20 hours after fabrication	NaOH (g./100 ml.)				
	Nil	0·0025	0·0025	1·0275	1·0275
	7	0·0520	0·0471	0·9724	0·8976
	14	0·0612	0·0546	1·0022	1·0026
	28	0·0788	0·0693	1·0275	1·0553
	KOH (g./100 ml.)				
	Nil	Trace	Trace	Trace	Trace
	28	0·1424	0·0740	0·0360	0·0500
Mortars placed in storage solutions after 28 days storage in moist air	NaOH (g./100 ml.)				
	7	0·0320	0·0304	..	
	KOH (g./100 ml.)				
	7	0·0466	0·0325	..	..

Conclusions are:—

(1) Alkalis were lost from all mortars which were stored in water. Mortars which contained reactive aggregate lost slightly less alkalis than those which contained non-reactive aggregate.

(2) After 28 days storage in water most of the alkalis were lost from the non-reactive aggregate mortar. During the same period most of the sodium hydroxide and about half of the potassium hydroxide were lost from the reactive aggregate mortar.

(3) Mortar stored in 1 per cent. sodium hydroxide solution at first appeared to take up sodium hydroxide from the storage solution, but later seemed to lose it again. The latter effect is probably due to some of the gel being extruded from the mortar and dispersed in the storage solution as small amounts of silica have been detected in it.

(4) The rate at which potassium hydroxide was lost from mortar was decreased by storing in 1 per cent. sodium hydroxide solution.

(iv) *Tensile Strength Changes of Mortars Stored under Various Conditions*

With the exception of the non-reactive rounded quartz sand from Paringa, South Australia, which was substituted for Leighton Buzzard sand, the materials and mixtures were the same as those used in sections (i), (ii) and (iii). Test bars and briquettes were stored under each of the following conditions: (*a*) in dry air; (*b*) in moist air; (*c*) in 1 per cent. sodium hydroxide solution; and (*d*) in water.

The relations between expansion and tensile strength of mortars at various ages stored under different conditions are shown in Table 5. Each recorded tensile strength, which is the mean of three determinations, is given to the nearest 10 lb. per sq. in. Differences between the maximum and minimum values within each group of three were generally lower for mortars containing reactive aggregate (less than 60 lb. per sq. in.) than for those containing non-reactive aggregate (50-110 lb. per sq. in.). Chance differences in the test pieces and testing procedure seem to cause these variations.

TABLE 5.—THE RELATIONS AT VARIOUS AGES BETWEEN EXPANSION AND TENSILE STRENGTH OF MORTARS CONTAINING NON-REACTIVE AND REACTIVE AGGREGATES WHEN STORED UNDER DIFFERENT CONDITIONS

Storage	Time after Fabrication	Non-reactive Aggregate		Reactive Aggregate	
		Expansion	Tensile Strength	Expansion	Tensile Strength
	days	%	lb./sq. in.	%	lb./sq. in.
In dry air	1	+0·000	120	±0·000	160
	6	−0·018	370	−0·016	370
	15	−0·041	530	−0·029	330
	22	−0·059	510	−0·040	330
	29	−0·073	470	−0·052	320
	36	−0·083	480	−0·065	330
	43	−0·090	510	−0·069	270
	57	−0·102	550	−0·081	290
	85	−0·121	600	−0·092	220
	113	−0·137	560	−0·108	230
	141	−0·163	530	−0·137	380
	169	−0·187	600	−0·155	260
	197	−0·194	590	−0·164	270
In moist air ..	1	±0·000	100	±0·000	170
	6	−0·007	360	+0·004	350
	11	−0·006	370	0·015	340
	16	−0;003	440	0·057	210
	23	−0·004	470	0·183	140
	30	−0·008	450	0·310	130
	44	−0·007	410	0·617	100
	58	−0·004	360	0·837	80
	86	−0·007	330	1·059	60
	114	−0·007	350	1·244	40
	142	−0·017	370	1·351	70
	170	−0·014	440	1·383	70
	198	−0·015	410	..	..

TABLE 5.—THE RELATIONS AT VARIOUS AGES BETWEEN EXPANSION AND TENSILE STRENGTH OF MORTARS CONTAINING NON-REACTIVE AND REACTIVE AGGREGATES WHEN STORED UNDER DIFFERENT CONDITIONS —*continued.*

Storage	Time after Fabrication	Non-reactive Aggregate		Reactive Aggregate	
		Expansion	Tensile Strength	Expansion	Tensile Strength
	days	%	lb./sq. in.	%	lb./sq. in.
In 1 per cent. sodium hydroxide solution	1	±0·000	110	±0·000	130
	6	+0·025	370	+0·007	380
	11	0·027	400	0·027	190
	15	0·030	420	0·077	160
	22	0·035	400	0·218	110
	29	0·034	370	0·393	150
	36	0·036	400	0·513	150
	43	0·038	380	0·577	160
	57	0·040	440	0·677	180
	85	0·043	380	0·824	160
	113	0·043	430	0·923	170
	141	0·044	410	1·028	200
	169	0·037	360	1·090	150
In water	1	±0·000	150	±0·000	180
	6	+0·002	310	+0·001	300
	11	0·006	360	0·015	190
	15	0·011	340	0·033	120
	22	0·013	370	0·097	120
	29	0·012	380	0·150	110
	43	0·016	350	0·219	90
	57	0·016	340	0·240	110
	85	0·018	350	0·262	120
	113	0·021	330	0·275	90
	141	0·021	310	0·279	120
	169	0·021	360	0·278	130
	197	0·022	330	0·280	150

Conclusions are:—

(1) Mortars containing non-reactive aggregate neither expanded significantly nor showed any abnormal decreases in tensile strength during storage under any of the conditions used.

(2) The expansions of reactive aggregate mortars were of the same order as those shown in Table 2. The tensile strengths of all these mortars decreased abnormally.

Observations on the mortars showed:—

(1) Mortars containing non-reactive aggregate developed neither reaction spots nor cracks.

(2) Mortars containing reactive aggregate developed reaction spots and cracks.

(3) In all storage conditions, cracks, which were associated with reacted aggregate particles, were first observed six days after fabrication, when mortar expansions were less than 0·01 per cent. These cracks, 'which were short and fine when first observed, lengthened and widened as the opal particles reacted and expanded.

Other observations confirmed those given in section (1).

4. Discussion

It will be shown in later papers that expansion depends (other things being equal) on three variables, namely, the relative amounts of alkalis, reactive material, and uncombined water. Mortars stored in dry air and in moist air differ in their amounts of uncombined water; those stored in alkali solutions and in water contain larger amounts of uncombined water than those stored in dry air and in moist air. The alkali contents of mortars stored in dry air and in moist air remain constant whereas those of mortars stored in alkali solutions and in water can undergo significant changes. It should be remembered that the following discussion only concerns small mortar test pieces. It is probable that only the surface layer of large concrete specimens or structures would be similarly affected by environmental conditions.

Depending on the amount available, uncombined water has two opposing actions on the expansion of reacting opal. Firstly, the volume of a reacting opal particle increases as it absorbs water. Consequently, within certain limits, mortars should expand by amounts depending on their uncombined water contents. Secondly, as reacting opal absorbs water, the properties of the reaction product change from those of a solid (gel) to those of a liquid (sol). The sol has a negligible expansive effect compared with the gel. Thus, for mortars which contain large amounts of uncombined water, the change in properties of the reaction product is rapid and expansions are not as high as those of mortars which contain normal amounts of uncombined water.

Provided the concentration of alkalis in the storage water or solution is lower than that in the solution in the mortar, alkalis will diffuse from the mortar into the surrounding medium. Thus small mortar specimens stored in water are unlikely to expand excessively as the amount of reaction product formed is small because of the decrease in the amount of alkalis available for reaction. Furthermore, since the amount of available water in the mortar is high the reaction product should form a sol rather than a gel.

When the storage medium is a sodium hydroxide solution (potassium hydroxide solution should behave similarly) sodium and hydroxyl ions will pass into or out of the mortar, depending on whether the concentration of these ions in the storage solution is greater or less than that in the solution in the mortar. If the concentrations of ions in the storage solution and mortar solution are approximately equal, opal will be attacked at a rate equivalent to that in mortars stored in moist air. It does not follow, however, that mortars stored in alkali solutions will expand at a rate equivalent to those stored in moist air since in the former large amounts of uncombined water are available for absorption by reacting opal, and the reaction product should form a sol rather than a gel.

A feature of reactive aggregate mortars when stored in water or 1 per cent. sodium hydroxide solution is that some of the reaction product is deposited as a cloudy gel on the mortar surfaces, but since these cloudy gels, which are very soft, are unlikely to expand the mortar, they will not be considered further here.

All mortars which contained reacted aggregate particles were cracked, although the expansions of those stored in dry air were negative. These observations indicate that mortar deterioration cannot always be detected by length changes of test bars. Tensile strength changes, however, revealed that all mortars containing reacted aggregate had deteriorated.

Cracks produced in mortars stored in moist air, in 1 per cent. sodium hydroxide solution, and in water were caused by the reacting particles exerting disruptive swelling pressures on the mortar and not by drying shrinkage, since the cement in these mortars retained much of their excess mixing water. In mortars stored in dry air cracks and reduction in tensile strength were due to the combined effect of the slight swelling of reacted particles caused by the absorption of some water and of the shrinkage of hydrated cement as it lost water. Since these are opposing actions, the resultant cracking is equivalent to that produced by a slightly greater swelling of reacted particles in a similar mortar in which the cement does not shrink abnormally (i.e. mortars stored in moist air).

5. Summary of Conclusions

(i) Absorption of water by reacting opal particles caused mortar expansion. Removal of uncombined mixing water from mortar inhibited expansion.

(ii) Cracks, associated with reacted opal particles, were observed in mortars stored under all conditions used.

(iii) Cracks reduced the tensile strength of all reactive aggregate mortars.

NOTES

"Australian Journal of Scientific Research"

In collaboration with the Australian National Research Council, C.S.I.R. has completed arrangements for the establishment in Australia of a new journal, as a medium for the publication of research papers of outstanding merit, open to receive contributions from research workers irrespective of the organization to which they are attached.

The journal, to be known as the *Australian Journal of Scientific Research*, will be printed in two series, both to be issued quarterly in the first instance. It is expected that the first number of Series A (Physical Sciences) will be issued in March 1948, and the first number of Series B (Biological Sciences, including agriculture) in February 1948.

Dr. N. S. Noble has been appointed as Editor and is stationed at Head Office. Editorial policy will be decided by an Editorial Board under the Chairmanship of the Editor and comprising as members Professors W. J. Dakin, E. J. Hartung, L. H. Martin, and J. G. Wood.

It is intended to maintain a high standard of quality by adhering to a strict refereeing system, and while members of the Board may referee some papers themselves, it is intended that where necessary the services of outside referees will be called upon. It is obvious that there can be no absolute standard of quality, but, as a general guide, the Board has decided that papers to be acceptable for publication in the new journal should be of such a quality that they would be acceptable to specialist journals overseas.

It is intended that the Council's Bulletin series will continue, and that the present C.S.I.R. Journal will be issued throughout 1948 at least.

Papers intended for the new journal should be submitted to Dr. Noble at the Council's Head Office, 314 Albert-street, East Melbourne.

The United Nations Educational, Scientific and Cultural Organization

The present fashion for the extensive use of initials may well cause difficulties for students of philology in years to come. The growth of the United Nations and its satellite organizations has produced a heavy crop of new symbols representing the complicated, high-sounding titles of various new bodies. During November, the annual conference of UNESCO will be held at Mexico City, and in recent months there have been various references in the daily Press to this body. What is UNESCO? What are its aims and objects? How does it function? What is its significance to Australians and, in particular, to Australian scientists? The purpose of this article is to give a brief description of its history, its present organization and activities, and its plans for the future.

The United Nations Educational, Scientific, and Cultural Organization is one of the specialized agencies of the United Nations, reporting directly to the Economic and Social Council. It grew out of the

Conference of Allied Ministers of Education which met in London at regular intervals from 1942 to 1945 to plan for the reconstruction of the educational facilities of occupied Europe. It soon became clear that the problems in this field which would await solution when victory was achieved would be enormous, and it was thought that an international organization for education would be desirable. The planning of such an organization to undertake the reconstruction of educational and cultural facilities in Europe was therefore commenced, and the work was stimulated by the discussions at San Francisco in 1945 when the United Nations Charter was drawn up, and it was agreed that the promotion of international cultural and educational cooperation should be an objective of the United Nations. Following on this, the British and French Governments invited all Governments of the United Nations to be represented at a Conference in London in November 1945 to establish an appropriate international body.

Forty-four Governments were represented at this Conference, and it was agreed that a United Nations Educational, Scientific, and Cultural Organization should be established. (The original proposal had been for a United Nations Educational and Cultural Organization; the inclusion of the word Scientific in the title was agreed to at the Conference largely as a result of the missionary efforts of Dr. Joseph Needham, F.R.S., who, during the time he was in charge of the Sino-British Scientific Cooperation Office in Chungking, had campaigned vigorously for greater organization in international scientific cooperation.)

The purposes and functions of UNESCO, as defined briefly in the final act of the Conference, are as follows:—

". . . to contribute to peace and security by promoting collaboration among the nations through education, science, and culture in order to further universal respect for justice, for the rule of law, and for the human rights of the world, without distinction of race, sex, language, or religion, by the Charter of the United Nations."

Its functions are: (*a*) to collaborate in the work of advancing mutual knowledge and understanding of peoples, through all means of mass communication; (*b*) to give fresh impulse to popular education and to spread culture; and (*c*) to maintain, increase, and diffuse knowledge. But "with a view to preserving the independence, integrity, and fruitful diversity of the cultures and educational systems of the States members of UNESCO, the Organization is prohibited from intervening in matters which are essentially within their domestic jurisdiction." These are wide and ambitious functions!

The Organization envisaged by the Conference consists of a General Conference, an Executive Board, and a Secretariat. A Preparatory Commission was also set up by the Conference to make detailed arrangements for the establishment of the Organization. Sir Alfred Zimmern, formerly Professor of International Relations at Oxford, was appointed as Secretary-General of the Preparatory Commission, and on his resignation he was succeeded by Dr. Julian Huxley, the eminent biologist. The Preparatory Commission met in June 1946, and at the first General Conference in Paris in November 1946 UNESCO came formally into being, with its headquarters in Paris.

The Organization has thus been in existence just over a year, during which time progress has been made in the building up of its Secretariat and in the planning and initiation of its activities. The cumbersome structure of international organizations, which seems inescapable when a large number of separate nation States attempt to cooperate, tends to attract undue attention to constitutions, acts, and organizational arrangements at the expense of the actual work being undertaken. UNESCO is no exception, but let us attempt to examine what the Organization is actually doing or proposing to do.

Apart from organizational difficulties, its activities are obviously limited by its budget. The General Conference in Paris agreed to a total budget for the current year (1947) of $6,950,000. Of this, however, $950,000 was to meet costs incurred prior to 1947, leaving $6,000,000 for UNESCO activities in 1947, not a very large sum for such high functions. The programme finally adopted by the Executive Board falls into two main groups of activities:—

    (a) Four large-scale projects of a comprehensive nature.

    (b) Specialized activities within the fields of education, science and culture.

In the first group comes the reconstruction and rehabilitation of educational, scientific, and cultural life in countries devastated by war, a project of great urgency in Europe and parts of Asia—an urgency which it is hard to grasp in Australia which has emerged from the war physically unscathed. Active field work in this project is at present being undertaken in Czechoslovakia, Greece, Poland, and Yugoslavia, as well as in Italy and Austria. In cooperation with other agencies and organizations a world-wide campaign is in progress for the collection of contributions in money, materials, and services for distribution in the devastated areas.

The second large-scale project has the ambitious and long-range object of teaching the illiterates of the world (who number more than one-half of its population) to read and write, and simultaneously campaigning for provision of a basic minimum of education for all the peoples of the world. Linked with this is the third project, that of education for international understanding which is of a continuing character seeking to stimulate and assist the active study of international relations in all countries.

The fourth large-scale project is definitely a scientific one, consisting of a far-reaching programme of research into the needs and possibilities of the vast forested Amazon area, which comprises four million square miles in the heart of the South American continent. A team of scientists making preliminary investigations includes a tropical botanist, an expert on tropical diseases, and two anthropologists. Conversations are being held with the Governments of Brazil, Bolivia, Colombia, Ecuador, Peru, Venezuela, Great Britain, France, the Netherlands, and the United States to examine the possibilities of establishing an International Research Institute at the mouth of the Amazon River.

So much for the large-scale projects. The specialized activities within the fields of education and culture can only be mentioned here. They include a study of educational techniques, the coordination, standardization, and improvement of educational statistics, a study of the tensions affecting international understanding, a preliminary study of the international aspects of philosophy and humanistic studies

as they are related to UNESCO's objectives, projects in the fields of literature and the theatre, music and the visual arts, studies of the needs and resources of libraries and museums, and studies on the use of mass communication media—films, radio, and the press.

Natural sciences play a major role in the reconstruction and rehabilitation project, particularly in relation to scientific and technical schools, colleges, universities and research institutes in war-devastated areas. The major activity being undertaken, however, is the establishment of Field Science Cooperation Offices in parts of the world which are remote from the main centres of science and technology. The object of these offices is to facilitate contact between scientists and technologists in various parts of the world, to assist in the solution of problems of scientific literature, translation, and reprints, and to assist the exchange of scientific men in the areas concerned. They will also serve as reception and distribution centres for scientific information, particularly of problems indigenous to the region. In addition to the office already established in the Hylean Amazon (at Rio de Janeiro) two more offices are located in the Far East and the Middle East. It is hoped to set up an office in Southern Asia in 1948.

The Natural Sciences Division of UNESCO is also engaged on a study of scientific documentation and scientific work of international significance. It is collecting information on scientific apparatus and scientific cinema films, is preparing a world register of scientists, and is investigating means of facilitating the travel of scientists throughout the world. It is also providing assistance for the already established International Scientific Unions, in an attempt to stimulate their activities by removing some of the factors which have hindered their work in the past. Office accommodation and secretarial staff for the International Council of Scientific Unions has been provided at UNESCO House in Paris.

From this necessarily sketchy outline of UNESCO activities it will be seen that the Organization is definitely attempting a number of practical tasks within the framework of its objectives; indeed some criticism has already been voiced that it is attempting to do too much and that it would be better if it concentrated its activities, in the first instance at any rate, on a smaller number of projects. Whether or not this criticism is justified remains to be seen. Much will depend on the calibre of the staff recruited and on the support given by member States to its activities whether this " magnificent experiment in international understanding " succeeds. The most notable absentee from the list of members is the U.S.S.R., but most other members of the United Nations have joined. It is interesting to note that UNESCO is not the first attempt at international cooperation in the cultural field, as the League of Nations established an International Institute of Intellectual Cooperation, with many of its objects similar to those of UNESCO. This organization did not flourish, however. The main reasons for its failure would appear to have been lack of financial resources and also the fact that it was insufficiently executive and practical. The proposed organization for UNESCO should avoid the latter difficulties. Whether the first difficulty can be avoided will depend on the support which the member States are prepared to give it.

UNESCO has recommended that in each member State there should be National Cooperating Bodies to maintain liaison between it and the country concerned. In Australia, the formation of such bodies

has been entrusted to the Commonwealth Office of Education. This office is the pivot in Australia for all UNESCO activities and, as UNESCO develops its work, more and more information should be available. It is hoped that all scientists in Australia and others interested in UNESCO's work will be prepared to support the activities of the National Cooperating Bodies in as practical a way as possible, by providing publicity for the work of UNESCO. This should then help to ensure the Organization's success, by enabling the individual to take an active interest in, and give support to, its activities.

"Prevention of Deterioration Abstracts"

The National Research Council of the National Academy of Sciences (Prevention of Deterioration Center, Room 204), 2101 Constitution-avenue, Washington, D.C., can now offer the *Prevention of Deterioration Abstracts* on a yearly subscription basis. These Abstracts are set up under the following headings: biological agents; electrical and electronic equipment; fungicides and other toxic compounds; JAN Deterioration Prevention Committee; lacquers, paints and varnishes; leather; lubricants; metals; miscellaneous; optical instruments and photographic equipment; packaging and storage; plastics, resins, rubbers, and waxes; textiles and cordage; wood and paper. Items abstracted include journal articles, patents, specifications, unpublished reports prepared by various Army, Navy and other governmental groups, and unpublished British, Australian, and Canadian reports.

There will be approximately 1,500 pages of the Abstracts per year. The individual abstracts are in loose leaf form, so that they may be arranged in the manner desired by the individual receiving them. Throughout the calendar year, all the abstracts classified under any one heading will be numbered consecutively.

Comments made by the personnel of the Prevention of Deterioration Center are added to many of these abstracts. In the comments attempts are made to relate a specific report with other relevant ones, to evaluate reports, or to make suggestions concerning further needed research.

The price, which includes two binders and index guides, will be $37.50 per year. Two binders are required for one year's subscription. The fiscal year will be from July 1 to June 30. The Abstract series started in April 1946 and back issues may be obtained at the regular subscription price.

Reviews

"ADVANCES IN GRASSLAND HUSBANDRY AND FODDER PRODUCTION: SECOND SYMPOSIUM."

(Bulletin 38 of the Imperial Bureau of Pastures and Field Crops, Aberystwyth, Wales, June 1947. 84 pp. Price 6s. (sterling). Obtainable from the Central Sales Branch, Imperial Agricultural Bureaux, Penglais, Aberystwyth, Wales.)

Bulletin 32 was the first symposium in this series. The two bulletins are akin to the earlier "Herbage Reviews" series.

The bulletin opens with four articles and the remainder of the "symposium" is a series of summaries, in some instances lengthy, of eleven papers and reports of diverse origin and objective.

The first article on objectives in veld investigations in Southern Rhodesia by West and Rattray gives an excellent appreciation of the problems of veld management and the techniques to be used in the investigations. The similarity of environment to coastal and sub-coastal Queensland gives added interest to the Australian reader. A short article on the economics of ley farming by A. W. Ashby follows. This is mainly of local interest. Professor Travin's discussion on " Formation of Plant Species " is an aspect of geo-botany which is not commonly included in an agricultural botany publication. Travin deduces a correlation between the process of species formation and seismic and volcanic activity and associates new species formation with regions of young geo-synclines. The statistical data presented are not particularly convincing. The fourth article is on " Forage production in Switzerland "—a translation by Miss Roseveare from the German by Alfred Kauter. This article traces the intensification of cropping and sown pastures for a period of ten years including the war period, largely because of the urgency of increasing home food production. The methods adopted included intensive extension work and some measure of compulsory ploughing and cropping.

The summaries of papers follow and include two of Australian origin, Schofield's study of coastal pastures at South Johnstone, Queensland, and Andrew's report on pasture investigations in Victoria during 1944/45. The other nine summaries are on a Scottish method of grassland improvement, agricultural seed production as a new industry in Welsh farming, grassland research and advisory work in Sweden, birdsfoot trefoil as a forage legume, hay dehydration in the south-western United States, U.S. Regional Pasture Research Laboratory, weeds in Fiji, pastures and fodder plants in Sierra Leone, and land resources of Tripolitania.

The subjects of this bulletin are both interesting and valuable but it is scarcely a symposium; rather is it a review.

J. G. D.

" CHEMICAL COMPOSITION OF PLANTS AS AN INDEX OF THEIR NUTRITIONAL STATUS."

By D. W. Goodall, Ph.D., D.I.C., F.L.S., and F. G. Gregory, D.Sc., F.R.S.

(Technical Communication 17 of the Imperial Bureau of Horticulture and Plantation Crops, East Malling, Kent, England, July, 1947. Pp. 167, bibl. 936. Price 9s. (sterling). Obtainable from Imperial Agricultural Bureaux, Central Sales Branch, Penglais, Aberystwyth, Wales.)

This communication may be divided roughly into three parts, which deal respectively with (i) theoretical aspects of the relationship of yield to nutrition supply, and the effect of this on concentration of nutrients in the plant; (ii) detailed consideration of the methods and techniques of plant sampling and analysis, their application, and the possible interpretations of the results; (iii) the comparison of plant analysis with field trials, soil analysis, and the use of deficiency symptoms as methods of nutritional diagnosis.

The publication should be of considerable assistance to the present-day worker on plant problems who must decide such questions as: Are field trials or pot cultures most suitable? Will the symptoms revealed by plant injection tell a sufficient story or must they be

backed by soil analysis, and if soil analysis is used, are chemical or biological methods preferable? Will observation of weed flora or of obvious deficiency symptoms give the final answer or can that be revealed only by determination of the most minute trace element in the plant? What have the hundreds of previous workers with their different methods and different techniques achieved? What is the lesson of their experience? What do their data signify?

In addition to the comprehensive bibliography of over 900 entries, subject and author indexes are provided.

Recent Publications of the Council

Since the last issue of this *Journal,* the following publications of the Council have been issued:—

> *Bulletin No.* 202.—" The Strain Complex and Symptom Variability of Tomato Spotted Wilt Virus," by D. O. Norris, M.Sc. (Agric.).

The great variation in symptoms produced by the spotted wilt virus has puzzled plant pathologists since the disease was first studied. The present work demonstrates that most of the variability within any one species is, in fact, due to the virus being not an entity but a complex of closely related strains. This is the fundamental cause of variation, but environment plays an important subsidiary part. There are at least five distinct strains, with symptoms varying in severity from a barely distinguishable mottle to complete necrosis.

A particular tomato plant may be infected by just one of these strains or by several different ones although commonly all five are present, the symptoms depending upon which strain predominates. The most severe strain, called the " tip-blight " strain, sometimes occurs on its own, when it produces a disease so severe that the young growing part of the plant is rapidly killed.

The tomato normally encourages the severe strains of the virus to multiply, but it might prove practicable to evolve a variety of tomato which would encourage only the milder strains of virus, thus reducing the severity of the disease to a point where it scarcely affects the yield.

> *Bulletin No.* 206.—" Pedogenesis Following the Dissection of Lateritic Regions in Southern Australia," by C. G. Stephens, M.Sc.

This Bulletin deals with the genesis and relationships of soils found in the various lateritic regions of southern Australia. It is shown that the different soils in any such region are related to the various horizons of the original lateritic soil and its parent rock. In addition it is demonstrated that parallel relationships exist between the soils and the original lateritic soil profile for all regions. These relationships are discussed for a number of areas where soil surveys have been carried out, and a diagram illustrating the connexion of the various named soil types and other features with the lateritic profile and with one another is presented. The diagram provides a

key to the interpretation of the soil and topographical pattern in lateritic regions in southern Australia and hence is of importance in ensuring sound soil survey procedure and in relating such surveys to fertility aspects of agricultural production in the areas concerned.

> *Bulletin No.* 218.—" Studies of the Physiology and Toxicology of Blowflies. 12. The Toxicity of DDT as a Contact and Stomach Poison for Larvae of *Lucilia cuprina.* 13. Insectary Tests of Repellents for the Australian Sheep Blowfly," by D. F. Waterhouse, M.Sc.

Tests described in this Bulletin show that, although DDT is not a particularly effective contact insecticide against prepupae, it is highly toxic in low concentrations when ingested, particularly by young larvae. It did not appear to produce any of the symptoms of nervous system distress which are so typical of its effect on adult blowflies and many other insects. The results indicate that if DDT were incorporated in a blowfly dressing it would be highly toxic to newly hatched larvae but older larvae would be less affected and eggs not at all.

In the tests of repellents, various essential oils and other materials were applied to sheep in an insectary to test their efficiency in preventing oviposition by *Lucilia cuprina.* The test materials were applied in a ring around cotton wool plugs, which had been soaked in an attractive solution and tied in the fleece. The repellent effect of Ceylon citronella oil was confirmed, but Java oil was not repellent. The oils of *Zieria smithii* and Huon pine were repellent, while oils from the leaf and wood of white cypress pine were ineffective. Of five eucalyptus oils, three were not repellent and two were attractive. Chemically pure oleic acid was repellent, while a commercial grade was not. Two materials containing indalone were effective repellents, as were also dimethyl phthalate and 612.

> " The Commercial Timbers of Australia—Their Properties and Uses," by I. H. Boas, M.Sc.

This book is divided into two parts. The first deals with the general technology of the timbers and discusses wood structure, including various forms of timber defect, the mechanical and physical properties of timbers, methods of seasoning, preservative treatment and durability, the bending of wood, grading, veneer and plywood, improved wood, the manufacture of paper from Australian hardwoods, and Australian essential oils.

The second part of the book lists the individual timbers, and gives the characteristics of each, including their density, the amount of shrinkage that can be expected, their durability, the seasoning required, main uses, and the availability of the timber. To aid in the selection of suitable timbers for specific purposes, the articles for which wood is commonly used are listed and the timbers that can be employed for each are given. A comprehensive bibliography dealing with Australian timbers is included, and the whole book has been carefully indexed.

(This book is not being distributed by C.S.I.R., but copies are obtainable from Tait Book Co. Pty. Ltd., 349 Collins-street, Melbourne. Price 12s. 6d., postage 8d.)

Forthcoming Publications of the Council.

At the present time, the following future publications of the Council are in the press:—

Bulletin No. 210.—" Preliminary Survey of the Natural Pastures of the New England District of New South Wales, and a General Discussion of Their Problems," by R. Roe, B.Sc. (Agric.).

Bulletin No. 212.—" The Frictional Properties of Lead-Base and Tin-Base Bearing Alloys: The Role of the Matrix and the Hard Particles," by D. Tabor, Ph.D.

Bulletin No. 213.—" Laboratory and Field Tests of Mosquito Repellents," by R. N. McCulloch, B.Sc., B.Sc.Agr., and D. F. Waterhouse, M.Sc.

Bulletin No. 214.—" The Preparation and Properties of Synthetic Cryolite," by P. Dixon, M.Sc., and T. R. Scott, M.Sc.

Bulletin No. 215.—" Studies in the Biology of the Skin and Fleece of Sheep. 4. The Hair Follicle Group and its Topographical Variations in the Skin of the Merino Fœtus," by H. B. Carter, B.V.Sc., and Margaret H. Hardy, M.Sc.

Bulletin No. 216.—" An Examination of the Peet-Grady Method for the Evaluation of Household Fly Sprays," by D. F. Waterhouse, M.Sc.

Bulletin No. 219.—" Spray Tests against Adult Mosquitoes. 1. Laboratory Spray Tests with Culicine (*Culex fatigans*) Adults," by D. F. Waterhouse, M.Sc. " 2. Spray Tests with Anopheline (*Anopheles punctualatus farauti*) Adults," by D. F. Waterhouse, M.Sc., and D. O. Atherton, M.Sc.Agr.

Bulletin No. 220.—" The Preparation and Use of Harvey's Reduced Strychnine Reagent in Oceanographical Chemistry," by D. Rochford, B.Sc.

Bulletin No. 221.—" Contributions to the Study of the Cell Wall. 4. The Nature of Intercellular Adhesion in Delignified Tissue. 5. The Occurrence, Structure, and Properties of Certain Cell Wall Deformations," by A. B. Wardrop, M.Sc., and H. E. Dadswell, D.Sc.

Bulletin No. 222.—" The Chaetognatha of South Eastern Australia," by J. M. Thomson, M.Sc.

Bulletin No. 223.—" Report of Marine Borer Survey in New Guinea Waters," by A. W. Shillinglaw, B.Sc., Dip.For., and D. D. Moore, B.Sc., A.S.T.C.

Bulletin No. 225.—" Studies on the Control of Wheat Insects by Dusts. 1. Field Tests of Various Mineral Dusts against Grain Weevils," by F. J. Gay, B.Sc., D.I.C., F. N. Ratcliffe, B.A., and R. N. McCulloch, B.Sc., B.Sc.Agr. " 2. Further Tests of Various Mineral Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C. " 3. The Use of Dust Barriers for the Control of Grain Insects," by F. J. Gay, B.Sc., D.I.C. " 4. The Use of DDT- and 666- impregnated Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C.

Bulletin No. 226.—" An Ecological Study of the Australian Plague Locust (*Chortoicetes terminifera* Walk.) in the Bogan-Macquarie Outbreak Area, N.S.W.," by L. R. Clark, M.Sc.

Bulletin No. 227.—" Studies on Perennial Veldt Grass (*Ehrharta calycina* Sm.)," by R. C. Rossiter, B.Sc.(Agric.).

Bulletin No. 228.—" Ecological Observations on the Small Plague Grasshopper, *Austroicetes cruciata* (Sauss.), in the Trangie District, Central Western New South Wales," by L. R. Clark, M.Sc.

Bulletin No. 229.—" Studies in Cement–Aggregate Reaction. I.—Australian Aggregates and Cements," by A. R. Alderman. D.Sc., Ph.D., A. J. Gaskin, M.Sc., R. H. Jones, B.Sc., and H. E. Vivian, B.Sc.Agr. " II.—The Effect of Alkali Movement in Hardened Mortar," by H. E. Vivian, B.Sc.Agr. " III.—The Effect of Void Space on Mortar Expansion," by H. E. Vivian, B.Sc.Agr. " IV.—The Effect of Expansion on the Tensile Strength of Mortar," by H. E. Vivian, B.Sc.Agr. " V.—The Effect of Void Space on the Tensile Strength Changes of Mortar," by H. E. Vivian, B.Sc.Agr. " VI.—The Effect of Carbon Dioxide," by A. J. Gaskin, M.Sc.

Bulletin No. 230.—" The Preparation and Properties of Aluminium Fluoride," by T. R. Scott, M.Sc.

Bulletin No. 231.—" Mint Weed, *Salvia reflexa* Hornem. Present Distribution and Status in Australia," by R. Roe, B.Sc.(Agric.), and N. H. Shaw, B.Agr.Sc.

Bulletin No. 232.—" Guide to the Medicinal and Poisonous Plants of Queensland," by L. J. Webb.

Bulletin No. 233.—" A Soil Survey of the Hundred of Seddon and Part of the Hundred of MacGillivray, Kangaroo Island, South Australia, including also Portions of the Hundreds of Cassini and Duncan," by K. H. Northcote, B.Agr.Sc., and B. M. Tucker, B.Sc.

Bulletin No. 234.—" Mineral Deficiency in Plants on the Soils of the Ninety-mile Plain in South Australia. Part 2," by D. S. Riceman, M.Sc., B.Agr.Sc.

Bulletin No. 235.—" The Algal Genus *Gracilaria* in Australia," by Valerie May, M.Sc.

" Handbook of Australian Pelagic Tunicates," by Harold Thompson, M.A., D.Sc.

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